



3 1176 00162 7927

NASA TM-82590

NASA Technical Memorandum 82590

NASA-TM-82590 19810016613

Tungsten Fiber Reinforced Superalloys—A Status Review

Donald W. Petrasek and Robert A. Signorelli

*Lewis Research Center
Cleveland, Ohio*

Prepared for the
Fifth Annual Conference on Composites
and Advanced Ceramic Materials
sponsored by the American Ceramic Society
Merritt Island, Florida, January 18-22, 1981

JUL 27 1981

NASA

TUNGSTEN FIBER REINFORCED SUPERALLOYS - A STATUS REVIEW

by Donald W. Petrasek and Robert A. Signorelli

National Aeronautics and Space Administration

Lewis Research Center

Cleveland, Ohio 44135

ABSTRACT

Improved performance of heat engines is largely dependent upon maximum cycle temperatures. Tungsten fiber reinforced superalloys (TFRS) are the first of a family of high temperature composites that offer the potential for significantly raising hot component operating temperatures and thus leading to improved heat engine performance. This status review of TFRS research emphasizes the promising property data developed to date, the status of TFRS composite airfoil fabrication technology, and the areas requiring more attention to assure their applicability to hot section components of aircraft gas-turbine engines.

INTRODUCTION

The key to the development of more efficient heat engines lies in the use of higher operating temperatures which in turn relies on the development of higher temperature materials. Heat engine efficiency is proportional to maximum cycle operating temperature. Increases in these operating temperatures have been accomplished through improvements in the properties of nickel and cobalt base superalloys as well as by the incorporation of sophisticated schemes for air cooling turbine components. The gap between the highest temperature at which superalloys can be used for long time service, 870° to 950° C (1600° to 1750° F) and the gas inlet temperatures for which engines are being designed, 1300°-1600° C (2370°-2910° F), is progressively widening. Thus, there is an increasing need to employ more air cooling in

the turbine section. However, as increasing amounts of compressor discharge air are diverted to cool hot-section components, the efficiency of a gas turbine engine drops. This coupled with the fact that nickel and cobalt base superalloys have been developed nearly to their technological limits, indicates that new types of materials are necessary to further raise engine efficiency. The challenge for materials engineering is to develop a new generation of high temperature materials which are qualified for long life application at material operating temperatures higher than 1000° C (1830° F).

The need for improved materials at elevated temperatures has stimulated research in many areas including efforts to develop fiber reinforced superalloy matrix composites. A number of fibers have been studied for such use including submicron diameter ceramic whiskers, continuous length ceramic filaments, boron filaments, carbon filaments, and refractory metal alloy wires. Attainment of high temperature strength with superalloy matrix composites using ceramic whiskers, continuous length ceramic filaments, boron filaments or carbon filaments as the reinforcing fiber have been unsuccessful to date. Boron and carbon react and are virtually dissolved when incorporated into superalloy materials. Attempts to develop coatings to prevent such reactions have been unsuccessful. Ceramic whiskers and continuous length ceramic filaments also have suffered from degraded fiber strength because of fiber-matrix reaction. The fiber-matrix reaction causes fiber surface flaws which act as local stress concentrations. These stress concentrations become nucleation sites for premature failure. The thermal expansion mismatch between ceramic fibers and typical candidate matrix alloys can induce severe stresses in composite materials. The combination of fiber surface roughening from reaction of the fiber with the matrix combined with thermally induced stress can cause catastrophic fracture of com-

posite test specimens even during fabrication. Because of the above factors, relatively low strength values have been achieved with such non-metallic fibers compared with those theoretically possible. However, due to the high use temperature potential of such composites, research efforts should and will continue in an attempt to overcome these problems.

The theoretical specific strength potential of refractory alloy fiber reinforced superalloys is less than that of ceramic fiber reinforced superalloys. However, the more ductile metal fiber systems are more tolerant of fiber-matrix reactions and thermal expansion mismatches. Also, the superalloy matrices can protect high strength refractory metal fibers from environmental attack. In laboratory tests, refractory fiber reinforced superalloy composites have demonstrated stress-rupture strengths significantly above those of the strongest superalloys. Tungsten fiber reinforced superalloy composites, in particular, are potentially useful as turbine blade materials because they have many desirable properties such as good stress-rupture and creep resistance, oxidation resistance, ductility, impact damage resistance, and microstructural stability. The potential of tungsten fiber reinforced superalloys (TFRS) has been recognized and has stimulated research to develop this material for use in heat engines. This status review of TFRS research emphasizes the promising property data developed to date, the status of TFRS composite airfoil fabrication technology, and the areas requiring more attention to assure their applicability to hot section components of aircraft gas-turbine engines. The approach will be to review first refractory metal fiber and matrix alloy development. This will be followed by a discussion of fabrication techniques for TFRS and property results of importance to their use at high temperatures. Specific criteria for employing TFRS as a turbine blade material are then presented. From

these criteria emerged a first generation TFRS Material, tungsten alloy fiber/FeCrAlY, which is currently under evaluation. Property, design, fabrication and fabrication cost data for this system are discussed in the final section.

Fiber Development

Refractory metal wires have received a great deal of attention as fiber reinforcement materials in spite of their poor oxidation resistance and high density. When used to reinforce a ductile and oxidation resistant matrix, they are protected from oxidation and their specific strength is much higher than that of superalloys at elevated temperatures. The majority of the studies conducted on refractory wire/superalloy composites have used tungsten or molybdenum wire, available as lamp filament or thermocouple wire, as the reinforcement material. These refractory alloys were not designed for use in composites nor for optimum mechanical properties in the temperature range of interest for thermal engine application, 1000°-1200° C (1830° to 2190° F). Lamp-filament wire such as 218CS tungsten was most extensively used in early studies. The stress-rupture properties of 218CS tungsten wire were superior to those of rod and bulk forms of tungsten and showed promise for use as reinforcement of superalloys. The need for stronger wire was recognized, and high strength tungsten, tantalum, molybdenum, and niobium alloys for which rod and/or sheet-fabrication procedures had already been developed were included in a wire fabrication and test program, (Refs. 1 to 4). The chemical compositions of these alloys are given in Table I. The above approach precluded development of new alloys specifically designed for strength at the intended composite use temperatures. The stress-rupture and tensile properties determined for the wires developed are summarized in

Table II and are compared with commercially available wire (218CS, W-1 percent ThO_2 and W-3Re).

Excellent progress was made in providing wires with increased strength compared to the strongest wires which were previously available. The ultimate tensile strengths obtained for the wires at 1093° and 1204° C (2000° and 2200° F) are plotted in Fig. 1. Tungsten alloy wires were fabricated having tensile strengths 2-1/2 times that obtained for 218CS tungsten wire. The strongest wire fabricated, W-Re-Hf-C, had a tensile strength of 2165 MN/m^2 (314 ksi) at 1093° C (2000° F) which is more than 6 times as strong as the strongest nickel or cobalt base superalloy. The ultimate tensile strength values obtained for the tungsten alloy wires were much higher than those obtained for molybdenum, tantalum or niobium alloy wire. When density is taken into account, the tungsten alloy wires show a decrease in advantage compared to tantalum, niobium or molybdenum wire, Fig. 2. Still the high strength tungsten alloy wires as well as molybdenum wires offered the most promise.

The elevated stress-rupture strength of such wire is more significant than the tensile strength since the intended use of the material is for long time applications. The 100 hour rupture strength at 1093° and 1204° C (2000° and 2200° F) is plotted for the various wire materials and compared to superalloys in Fig. 3. The rupture strength of tungsten alloy fibers was increased by a factor of 3 at 1093° C (2000° F) from about 434 MN/m^2 (63 ksi) for 218CS tungsten to 1413 MN/m^2 (205 ksi) for W-Re-Hf-C wire. The tungsten alloy wire was superior in stress-rupture strength to the other refractory wire materials with the exception of a tantalum alloy, ASTAR 811C, which was stronger than most of the tungsten alloy materials at 1093° C (2000° F). The strongest tungsten alloy wire, W-Re-Hf-C, was over

16 times as strong as superalloys at 1093° C (2000° F). The ratio of the 100 hour rupture strength to density for refractory metal wires and superalloys is plotted in Fig. 4. Again the stronger tungsten wire materials are superior to the other refractory metal wires. When density is taken into account the strongest tungsten wire material, W-Re-Hf-C, is more than 7 times as strong as the strongest superalloys at 1093° C (2000° F) and more than 13 times as strong at 1204° C (2200° F).

The processing schedules used to fabricate the newer high strength wires were not optimized to provide maximum strength at 1093° and 1204° C (2000° and 2200° F). Much more work is needed to maximize their properties. Considerable opportunity exists to develop wire processing schedules tailored for fiber-matrix composite use. The eventual application of TFRS composites will justify the added effort to further improve wire properties.

Matrix-Alloy Development

The matrix is the exposed component of fiber reinforced composites and therefore must be able to withstand high temperatures and an environment which can result in catastrophic oxidation and hot corrosion. The primary function of the matrix is to bind the fibers into a useful body and to protect the fibers from oxidation and hot corrosion. The matrix must be relatively ductile compared to the fibers to facilitate load transfer from the matrix to the fiber. It also must be capable of evenly redistributing local stress concentrations and resisting abrasion and impact damage from foreign objects. The matrix and reinforcing fiber must be able to co-exist without mutually induced disintegration that can result from chemical interactions that can degrade both the fiber and matrix properties. The most important factor in the initial selection of matrix composition is the ability of the

matrix to form a good bond with the fiber without excessive reaction occurring which could degrade the fibers properties.

For high temperature use, nickel-base, cobalt-base and iron-base superalloys are preferred as the matrices for refractory metal fiber composites because they have demonstrated strength and ductility at elevated temperatures as well as good oxidation and hot corrosion resistance.

A large proportion of the research effort conducted on refractory fiber composites has been on fiber-matrix compatibility with an effort being made to develop structurally stable composites by choosing a matrix composition which does not severely degrade the properties of the reinforcing fiber.

One of the first systematic examinations to determine the effect of alloying reactions on the strength and microstructure of refractory metal fiber composites was reported in Ref. 5 where copper-based binary alloys were used as a matrix for tungsten-fiber composites. The effects of alloying element additions to copper on the strength and microstructure of tungsten fiber composites were compared with mutually insoluble pure copper matrix composites exposed under the same conditions. The alloying elements studied were aluminum, chromium, cobalt, niobium, nickel, zirconium and titanium. Data obtained for solute elements in this system can be related to the expected behavior of these same elements in superalloys. These effects served as the basis for modifying superalloy matrix composition to control fiber-matrix reaction. Three types of fiber-matrix reaction were found to occur:

(1) diffusion-penetration reaction accompanied by a recrystallization of a peripheral zone of the tungsten fiber (2) precipitation of a second phase with no accompanying recrystallization (3) a solid solution reaction with no accompanying recrystallization in the fiber. Peripheral recrystallization was caused by diffusion of cobalt, aluminum, or nickel into the tungsten

wire. Compound formation occurred with titanium and zirconium. Chromium and niobium in copper formed a solid solution with tungsten with no accompanying recrystallization of the tungsten fiber. The greatest damage to composite properties occurred with the penetration-recrystallization reaction while the two-phase and solid solution reactions caused relatively little damage. Recrystallization of tungsten fibers in a Cu-10 percent Ni matrix is shown in Fig. 5.

A number of studies have been conducted on nickel induced recrystallization of tungsten fibers, Refs. 6 to 8. Recrystallization could be induced at low temperature by the presence of solid nickel on the surface of the tungsten wire. Once initiated, nickel-induced recrystallization required a continued source of nickel for propagation of the recrystallization front. Work reported in Ref. 6 found that palladium, aluminum, manganese, platinum and iron also greatly lowered the recrystallization temperature of tungsten. Based on such findings, superalloy matrix compositions were developed that caused limited reaction with the fiber and minimal fiber-property loss on work reported in Ref. 9. These superalloys contained high weight percentages of refractory metals to reduce diffusion penetration of nickel into tungsten. Additions of Ti and Al to the matrix were also made to form intermetallic compounds which would further reduce the diffusion of nickel into tungsten. A typical alloy that was developed was Ni-25W-15Cr-2Al-2Ti. The fiber stress to cause rupture in 100 hours at 1090° C (2000° F) was reduced only 10 percent in the composite compared to the equivalent fiber rupture strength tested in a vacuum outside a composite.

The problem of obtaining structure-stable composite materials from the nickel-tungsten and nickel-molybdenum systems were further examined in Ref. 10. These experiments showed that in reinforced metal composite mate-

rials in which the matrix and fiber form restricted solid solutions in the absence of intermetallic compounds, minimal fiber dissolution can be achieved by alloying the matrix with the fiber material up to a concentration that is close to the solubility limit. However, when the matrix and the fiber react to form intermetallic compounds, matrix saturation is not effective in controlling fiber attack by dissolution.

The effect of the composition of nickel and cobalt alloys on the structural stability of composite materials reinforced with tungsten fibers was determined in Ref. 11. The rate of interaction between the fiber and the matrix was determined from the extent of recrystallization of the fiber; the formation of intermediate phases at the interface; the solution of the fiber in the matrix and the formation of diffusional porosity. In those composites having a nickel matrix, a zone of accelerated recrystallization of the fiber was observed which increased with annealing time. The recrystallization rate of the fiber was highest with a matrix of pure nickel and somewhat lower with a nickel-chromium matrix. Alloying of nickel-chromium with iron, titanium, aluminum, and especially tungsten and niobium slowed down the recrystallization process considerably. The stability of the fibrous structure of the tungsten fiber was highest in a matrix of cobalt or cobalt alloys. The accelerated recrystallization of tungsten fibers in a matrix of nickel-cobalt was due to the presence of nickel. Unfortunately intensive solution of tungsten fibers occurred in a matrix of cobalt alloys and especially in pure cobalt.

Reaction of tungsten fibers with cobalt-base matrices was also determined in Ref. 12. Fiber-reinforced binary and multicomponent cobalt-base alloys were found to have a strong propensity to intermetallic compound formation at the matrix fiber interface. Four cobalt-base alloys were

identified that did not react with tungsten fibers after exposure for 1 hour at 1300° C (2370° F).

Work reported in Ref. 13 on the reaction of tungsten fibers with iron-base matrices concluded that certain such alloys can be successfully employed as matrices for composite materials reinforced with fibers of tungsten and its alloys. The matrix composition can be chosen so that it forms no intermetallic compounds with the tungsten fibers and the fibrous structure of the drawn tungsten wire is not affected by heating to 1300° C (2370° F). In most of the binary alloys investigated no recrystallization of the tungsten fibers occurred during annealing for 1 hour at 1300° C (2370° F). Dissolution of the fibers in the matrices was detected only rarely. However, in the majority of the matrices studied, layers of intermetallic compounds formed at the matrix-fiber interface. In the case of binary iron matrices alloyed with 5 and 10 percent aluminum no reactions were observed. After annealing for 1 hour at 1300° C (2370° F) no intermetallic compounds were formed in the binary iron matrices alloyed with 5-10 percent aluminum, 10 percent silicon, or 25 percent titanium, molybdenum, or nickel. The presence of nickel in multicomponent iron-base alloy matrix reinforced tungsten fiber composites accelerates the recrystallization of the fibers while increasing the amount of chromium and zirconium inhibits recrystallization. Complex alloying therefore offers a means of suppressing fiber recrystallization and the formation of intermetallic compounds at the interface between tungsten fibers and the iron-base matrix. Studies conducted on the reaction between tungsten fibers and a matrix of Fe-24Cr-5Al-1Y, Ref. 14, showed that only 10 percent of a 0.38-mm (0.015 in.) diameter fiber was reacted after exposure for 1000 hours at 1090° C (2000° F).

The reaction of tungsten fibers with binary alloys of iron, nickel and cobalt alloyed with 5, 10, or 25 weight percent of one of the following elements; aluminum, copper, silicon, zirconium, niobium, molybdenum, or tungsten were further investigated in Ref. 15. The composite samples were annealed at 1200°-1400° C (2190°-2550° F) for 1 hour. The interaction between the fiber and matrix was investigated by metallographic analysis and by change in hardness. The results of this study for samples annealed at 1200° and 1300° C (2190° and 2370° F) are summarized in Table III. Shown in Table III are the number of compositions investigated for each matrix system and the relative number of cases, based on a percentage, that resulted in the following reactions with the tungsten fiber, recrystallization, formation of an intermetallic compound, a diffusional penetration into the fiber and no detectable recrystallization or reaction with the fiber. At 1200° and 1300° C (2190° and 2370° F) contact between the tungsten fiber and the nickel binary alloys almost always induced recrystallization of the fiber. Nickel binary alloys containing 25 percent aluminum and 25 percent copper did not cause recrystallization of the fibers. As can be seen in Table III, the interaction of the tungsten fibers with binary alloys of cobalt generally did not lead to recrystallization of the fibers. At all annealing temperatures, however, a layer of intermetallic compounds was formed at the fiber-matrix interface. This process was suppressed at 1200° and 1300° C (2190° and 2370° F) only by alloying of the cobalt matrix with aluminum. Alloying of cobalt with nickel induced recrystallization of the fibers in addition to the formation of intermetallic compounds. The tungsten fiber was not recrystallized in most of the iron binary alloys studied. There was no interaction of any kind at 1200° C (2190° F) with alloys of iron and aluminum, silicon, titanium (10 and 25 percent), zirconium, niobium, chro-

mium (10 and 25 percent), molybdenum, and tungsten. An intermetallic layer was formed in most of the iron alloys at the fiber-matrix interface at 1300° C (2370° F). At 1300° C (2370° F) no interaction occurred only in the case of the matrix with 5-25 percent aluminum or 25 percent titanium.

The results of the preceding investigations show that selective alloying additions to nickel-base, cobalt-base and iron-base matrix materials offer a means of suppressing fiber recrystallization and the formation of intermetallic compounds at the fiber-matrix interface. A number of matrix compositions have been identified, particularly for iron base alloys, in which no detectable reaction occurs with tungsten fibers after short time exposures at temperatures up to 1200° C (2190° F).

The selection of matrix composition based on compatibility requires a compromise between two opposing requirements. A strongly bonded interface must be formed between the matrix and fiber to allow efficient stress transfer and maintain continuity during heating-cooling cycles, while at the same time a destructive reaction must be prevented while at the operating temperature. The first requirement implies the initiation of a chemical reaction, while the second requirement involves the prevention of chemical reaction. A continuing reaction at the operating temperature would be acceptable if the rate of reaction is sufficiently slow to give an adequate service life. Studies conducted on the rate of reaction between tungsten fibers and nickel-base, cobalt-base and iron-base alloy matrix materials indicate that compositions can be obtained satisfying the above requirements.

Most of the matrix compositions investigated that resulted in minimum reaction with the fibers involved the formation of intermetallic compounds which served to reduce interdiffusion. Thus, intermetallics might be sought as a natural occurring diffusion barrier. Use of a suitable protective

barrier between the fiber and matrix offers the possibility of a wider range of composition selection for composites for high temperature application. However, as pointed out in Ref. 16, the introduction of a second interface and a deposited coating, whose possible breakdown in service at high temperatures would cause a catastrophic decrease in strength, is not an attractive proposition to engine manufacturers and operators. It was pointed out in Ref. 17 that although diffusion barrier coatings on reinforcing wire are a potentially effective way to achieve control of fiber-matrix interaction, techniques attempted to date have not resulted in reproducible, successful barrier coatings for refractory alloy wire. Optimism continues, however, that such natural or deposited coatings are possible and will offer increases in both strength and use temperature. Trade-offs in compound composition and ductility offer a fruitful area for continued studies.

Sufficient evidence has been accumulated on matrix-fiber reactions to project that matrix alloy compositions with chemical compatibility and adequate bonding can be obtained with iron base, nickel base and cobalt base alloy systems. However, as will be discussed later in this paper, several additional requirements must be satisfied in selecting a matrix alloy, including ductility, thermal expansion compatibility, creep strength, strain hardening rate, oxidation and corrosion resistance and ease of fabrication. The basis for selecting matrix compositions to fill the host of requirements is in an early state of development. The approach taken in the NASA-Lewis program has been to select alloys with chemical compatibility to control fiber-matrix reactions and with ductility and low strain hardening to resist cracking from thermally induced cyclic stresses. Other requirements have been given consideration but were subordinated to those two needs. Variation of matrix composition within the composite geometry has also been used

to better satisfy the variation in requirements within a component. For example, a higher creep strength material with less thermal strain and lower oxidation resistance at moderate temperatures can be used in the base portion of a blade because it is a better match to fill the requirements in this portion of the component. Matrix alloy selection is a complex activity and much more experience is needed before guidelines can be established. Matrix-fiber reaction is one requirement that can be met, but the challenge is to satisfy simultaneously the many additional needs.

Composite Fabrication

The fabrication of matrix and fibers into a composite with useful properties is one of the most difficult task in developing refractory-wire-reinforced superalloys. Fabrication methods for refractory-wire-superalloy composites must be considered to be in the laboratory phase of development. Production techniques for fabrication of large numbers of specimens for extensive property characterizations have not yet been developed.

Fabrication methods can be classified as either solid phase or liquid phase depending upon the condition of the matrix phase during its penetration into a fibrous bundle. Liquid phase methods consists of casting the molten matrix using investment casting techniques so that the matrix infiltrates the bundle of fibers in the form of parallel stacks or mats. The molten metal must wet the fibers, form a chemical bond and yet be controlled so as not to degrade the fibers by dissolution, reaction or recrystallization. This control is difficult at very high temperatures since liquid metal temperatures of 1500° C (2730° F) are involved and contact time at temperature must be less than 1 minute, Ref. 18. Larger fiber diameters have been used to increase the size of the unreacted wire core and matrix alloy composition have been selected to reduce solute diffusion into the

fibers. Liquid phase methods are particularly suited to the preparation of fiber reinforced superalloys for gas turbine application because casting is the basic fabrication technique presently used for turbine blades and vanes. The technology developed for casting cooled blades via shell molds containing silica inserts could be applied directly to the fabrication of a reinforced blade, Ref. 16. Experience has shown, however, that the use of fibers having a diameter less than 0.075 cm (0.030 in.) in composites prepared by liquid metal infiltration leads to displacement of the wires. This is explained by the lack of rigidity of the unsupported span of the wire permitting surface tension to pull the fibers into a tight bundle near the mid-span, Ref. 16. Thus, control of alignment and spatial distribution of fibers at low volume fractions is difficult. A similar technique was attempted in Ref. 19 to cast tungsten fiber reinforced cobalt-base alloy test specimens. It was reported, however, that the matrix composition inside the fiber bundle became richer in tungsten than the matrix surrounding the fiber bundle and there appeared to be separation of the filament bundle from the matrix. An alternate means of using liquid-state technology is to coat the wire with the matrix by passing it as single strand or small bundles rapidly through a molten bath of matrix. The coated wires could then be diffusion bonded in closed dies to form the composite component.

Solid phase processing requires diffusion, which is time-temperature dependent. Solid phase processing temperatures are much lower than liquid phase processing temperatures; diffusion rates are much lower and reaction with the fiber can be less severe. The prerequisite for solid state processing is that the matrix be in either sheet, foil or powder form. Hot pressing or cold pressing followed by sintering is used for consolidation of the matrix and fiber into a composite component.

Use of matrix materials in the form of sheet or foil involves placing the reinforcing fibers between layers of the matrix sheet or foil which are then pressed together. They may be hot pressed or alternately cold pressed followed by diffusion bonding. An example of this type of processing is reported in Ref. 20. One of the most promising methods of manufacture of composite sheet materials is that of vacuum hot rolling, which gives high productivity and enables large sized sheets to be manufactured. In Ref. 21 a study was reported of the processing parameters for the manufacture of composite sheet material by vacuum hot rolling. A pack of several layers of tungsten fibers were placed in alternate layers between matrix alloy sheets; the pack was wrapped up in nickel foil, covered with thin mica sheets; and placed in a carbon steel sheath. The sheath was sealed by gas welding. The resultant pack was evacuated, heated and rolled. On the basis of the results obtained in this study the authors claim that it is possible to select rolling conditions ensuring maximum high-temperature strength in the resultant material, adequate adhesion between the fibers and matrix, freedom from porosity and strong welding between matrix sheets in the composite. A disadvantage of the use of thin sheet or foil is that most high strength superalloys are not available in this form, although economics rather than technical limitations cause the problem.

The powder metallurgy approach is one of the most versatile methods for producing refractory fiber-superalloy composites and has yielded some excellent results. Almost all alloy metals can be produced in powder form. However, the large surface area of the fine powders is easily contaminated and introduces impurities that must be removed. High capital cost equipment is necessary to apply pressure and temperature in an inert atmosphere. Most powder-fabrication techniques limit fiber content to 40-50 volume percent.

Despite these disadvantages, powder processing has been used to achieve control of matrix-fiber reactions and has resulted in excellent composite properties.

Slip casting of metal alloy powders around bundles of fibers followed by sintering and hot pressing was developed for the solid state fabrication of refractory fiber-superalloy composites, Ref. 9. The slipcast slurries consisted of a mixture of powders and an organic gel in water. Slurries were converted in a vibratory mold to a solid "green" composite which was sintered using a heating schedule designed to drive off the organic binder material and sinter the powders and fiber together without oxidizing either. The sintered composite was then isostatically hot pressed to full density. This method is capable of achieving good matrix consolidation and bonding between fiber and matrix without excursions into the liquid metal region which would greatly increase fiber matrix reactions. Although the slip-casting + sintering + hot pressing technique has demonstrated excellent success for uniaxially reinforced specimens, it is not regarded as an ideal method for component fabrication because most applications require some cross-ply fiber orientation, which is not easily accomplished with slip casting. Control of fiber distribution was also found to a problem using the slip casting technique.

In Ref. 22 a fabrication procedure was developed utilizing solid phase processing in which fiber distribution, alignment and fiber-matrix reaction could be accurately controlled. Matrix alloy powders were blended with a small quantity of organic binder (Teflon) and warm rolled into high density sheets. During rolling the Teflon formed an interlocking network of fibers which held the powder particles together. Fiber mats were made by winding the fibers on a drum, and then spraying them with a binder. The fiber array

was cut from the drum and flattened to form a fiber mat. Precollimated fibers in mat form were sandwiched between layers of matrix powder sheet and the matrix was densified and extruded between fibers by hot pressing. Fiber-matrix and matrix-matrix metallurgical bonding was achieved while preserving uniform fiber distribution and eliminating any voids. This procedure resulted in the fabrication of a single layer of fibers contained in the matrix material which was termed a monotape. Monotapes can be cut into any shape desired with any orientation of fiber desired and subsequently stacked up and hot pressed into a desired component. This approach appears to be the most viable method currently available for the fabrication of tungsten fiber reinforced superalloy composite components for use in heat engines.

Composite Properties

The principal reason for most of the work on refractory fiber superalloy composites has been to produce a material capable of operation as highly stressed components such as turbine blades in advanced aircraft and industrial gas turbine engines at temperatures of 1100° to 1200° C (2010° to 2190° F) or higher. Such an increase in temperature above the current limit of about 950° C (1740° F) for superalloys would permit higher turbine inlet temperatures, and markedly decreased cooling, thus improving engine performance and efficiency. An increase in blade temperature of 50° C (90° F) over current limits would be considered a significant improvement, Ref. 18. A review of gas turbine blade material property requirements such as presented in Refs. 23-26 indicates creep resistance, stress-rupture strength, low cycle fatigue, thermal fatigue resistance, impact strength, and oxidation resistance as properties of primary concern for turbine blade applica-

tion. The following section reviews the results obtained for refractory fiber/superalloy composites for the above property areas.

Stress-Rupture Strength

At temperatures of 1100° C (2010° F) and above, a superalloy matrix contributes very little to the rupture strength of the composite compared to the contribution of the refractory fibers. Fiber stress-rupture strength, volume fraction of fiber and the degree of fiber-matrix reaction all control the stress-rupture strength of the composite. Figure 6 is a plot comparing the 100 hour rupture strength at 1093° C (2000° F) for various fibers and composites containing 70 volume percent of these fibers, from studies conducted in Refs. 9, 27, and 28. The matrix composition (Ni-15Cr-25W-2Al-2Ti) was the same for all of the composites and as indicated in the plot, the stronger the fiber the greater the stress-rupture strength of the composite. The effect of fiber content on the stress-rupture strength of a composite is shown in Fig. 7, (Ref. 29). Stress-rupture strength increases linearly as the fiber content increases.

A comparison of the 100 hour rupture strength at 1093° C (2000° F) for some of the composite systems that have been investigated Refs. 9, 14, 19, 27, 28, 29, 30, 31 and 32 are given in Table IV and plotted in Fig. 8. Where possible, comparisons were made for composites containing 40 volume percent fiber. It should be noted that higher values would be obtained for these composite systems if the fiber content was increased. Also shown in the plot are the values for the 100 hour rupture strength for unreinforced alloys and for the strongest commercially available superalloys. The 100 hour stress-rupture strength of all of the alloys investigated was substantially increased by the addition of tungsten fibers. All of the 40 volume percent fiber composites had a 100 hour rupture strength greater than

that for the strongest commercially available superalloys. The W-Hf-C fiber composite system is the strongest composite system obtained to date. A 40 volume percent W-Hf-C fiber content superalloy composite is over 3 and 1/2 times as strong in rupture for 100 hours at 1100° C (2010° F) as the strongest commercially available superalloys. The composite containing a larger amount of fiber reinforcement (56 volume percent W-1 percent ThO₂ wire) in FeCrAlY also had an impressive stress-rupture strength, over 2 and 1/2 times that for the strongest commercially available superalloys.

The density of these composite materials is greater than that of superalloys and this factor must be taken into consideration. The stresses in turbine blades, for example, are a result of centrifugal loading; therefore, the density of the material is important. A comparison of the specific strength properties of composites and superalloys is therefore significant. Figure 9 is a plot comparing the ratio of the 1100° C (2010° F) 100 hour rupture strength to density for composites and superalloys. Even when density is taken into account, the stronger composites are much superior to the strongest commercially available superalloys. The composite containing 40 volume percent W-Hf-C wire is almost 2 and 1/2 times as strong as the strongest superalloys.

The comparison of stress-rupture strength between composites and superalloys is even more favorable for the composite when long application times are involved. Figure 10 is a plot of stress to rupture versus time to rupture for three different fiber compositions, each having the same matrix material, compared to the strongest superalloys. All of the fiber composite systems are stronger relative to superalloys for rupture in 1000 hours than for rupture in 100 hours at 1093° C (2000° F). The stress (to cause rupture) to density ratio versus time to rupture is plotted in Fig. 11. The

specific stress-rupture strength advantage for the composite also increases with time to rupture. The 40 volume percent tungsten fiber composite, for example, has about the same specific (density corrected) strength for rupture in 100 hours compared to superalloys but is almost twice as strong as superalloys for rupture in 1000 hours. For currently required blade lives of 5,000 - 10,000 hours this advantage becomes even greater.

A comparison of the range of values for the 100 hour rupture strength for tungsten fiber reinforced superalloy composites tested at 1093° C (2000° F) with the range for the stronger cast superalloys as a function of temperature is shown in Fig. 12. The strongest TFRS composite has the same rupture strength at 1093° C (2000° F) as does the strongest superalloy at 915° C (1680° F). This represents a material use temperature advantage for the composite of 145° C (320° F) compared to the strongest superalloy.

Figure 13 shows the density corrected values for rupture in 100 hours as a function of temperature. When density is taken into consideration the composite has a material use temperature advantage of 110° C (200° F) over the strongest superalloys.

Creep Resistance

The creep-rupture properties of Nimocast 713C reinforced with tungsten or tungsten-5 percent rhenium wire were evaluated and compared with the data determined for vacuum-cast Nimocast 713C in Ref. 31. Typical composite creep curves are shown in Fig. 14 together with a comparative curve for the unreinforced matrix. The creep curves for both materials exhibit the three characteristic stages of creep associated with conventional materials. Essentially, reinforcement reduces the second stage minimum creep rate markedly for a given applied stress due the presence of the more creep resistant fibers. The reduction in minimum creep rates observed on rein-

forcing Nimocast 713C suggests that the stronger, more creep resistant component, the fiber, controls the creep behavior. The lack of evidence of creep deformation in the matrix of the composite, except at the matrix-fiber interface adjacent to the fracture surface, also suggested that the behavior is controlled by the reinforcement. Similar results were obtained with tungsten-1 percent ThO₂ reinforced Hastelloy X composites, Ref. 33, tungsten-1 percent ThO₂ reinforced FeCrAlY composites, Ref. 14, and with tungsten-nickel composites, Ref. 34.

Fatigue

High-temperature materials in gas turbines are subject to cyclic stresses and strains. These can lead to the development of cracks and failures which conventionally are discussed in three separate groupings, depending on the magnitude and cause of the stresses:

High cycle fatigue

Low cycle fatigue

Thermal fatigue

a. High Cycle Fatigue

High Cycle fatigue failures can occur due to cyclic mechanical loads that lead to small scale yielding (for example, applied stresses which cause only localized plastic deformation while the rest of the structure is loaded in the elastic range). High cycle fatigue is often simulated in the laboratory by testing specimens under cyclic loads, the results being plotted as an S-N curve in which the applied cyclic stress amplitude (S) is plotted as a function of the number of cycles to failure (N). Typically, the number of cycles to failure is greater than 10⁵. Rarely are tests carried beyond 5x10⁸ cycles, while turbine blades in service may experience 10¹² or more cycles, Ref. 35.

High cycle fatigue tests have been conducted on W-1 percent ThO_2 /Hastelloy X composite specimens (Ref. 33). Fatigue tests were performed at 10 to 15 Hz using direct stress, tension-tension, axially loaded specimens. The specimens were cycled from a minimum stress to a selected maximum stress and back to the minimum stress. The load ratio, R , ranged from 0.1 to 0.7 for unreinforced Hastelloy X and 0.1 to 0.2 for the composite. The composite load ratios represented a more severe test. The stress ratio, A , ranged from 0.16-0.82 for unreinforced Hastelloy X and 0.52-0.91 for the composite. Test temperatures were room temperature, 816° , 900° and 980° C (1500° , 1650° , and 1800° F). Composite specimens were unidirectionally reinforced and contained 23, 30, and 37 volume percent fibers. The stress to cause failure in 1×10^6 cycles versus temperature is plotted in Fig. 15. Unreinforced Hastelloy X data are plotted for comparison. The composites were stronger at all temperatures, ranging from 1.2 times as strong at room temperature to 4 times as strong at 980° C (1800° F). The ratio of fatigue strength to ultimate tensile strength for the same materials is plotted in Fig. 16. For all test temperatures, the ratio for the composite was higher than that for the Hastelloy X, indicating that high cycle fatigue resistance of the composite is controlled by the fiber.

The high cycle fatigue strength for W-1 percent ThO_2 /FeCrAlY composites was determined at 760° and 1038° C (1400° and 1900° F) in Ref. 36. Fatigue tests were performed at 30 Hz using direct tension-tension, axially loaded specimens. The specimens were cycled from a minimum stress to a maximum stress and back to the minimum stress. The load ratio, R , was 0.3 and the stress ratio, A , was 0.5. Composite specimens containing 20, 35, and 40 volume percent fibers were tested. Figure 17 is a plot of maximum stress versus the number of cycles to failure for specimens tested at 760° C

(1400° F). The maximum stress versus number of cycles to failure for specimens tested at 1038° C (1900° F) is plotted in Fig. 18. The results again indicate that fatigue is controlled by the fiber. The composite containing 40 volume percent fiber had a 1×10^6 cycle fatigue strength to ultimate tensile strength ratio of 0.9, at 760° C (1400° F). Similar ratios for some superalloys range from 0.52 to 0.67 at the same test temperature, Ref. 37. Thus the composites' response to high cycle fatigue appears to be superior to superalloys. Figure 19 is a plot of the 1×10^6 cycle fatigue strength to ultimate tensile strength ratio for some superalloys and the range of values obtained for TFRS composites showing the advantage for the composite. Push/pull and reverse bend fatigue strength data were determined for a W/superalloy composite in Ref. 29. The fatigue strength measured in push/pull tests at 20°, 300°, and 500° C (70°, 570°, and 930° F) was substantially increased by the introduction of 40 volume percent tungsten wires. With cantilever specimens tested in reverse bending, a significant increase in fatigue strength also resulted from the incorporation of tungsten wires.

b. Low Cycle Fatigue

Low cycle fatigue is characterized by high cyclic loads that lead to failure after a relatively small number of load cycles, usually less than 10^4 to 10^5 . Fracture under low cycle fatigue conditions is generally associated with largescale yielding.

Limited work has been reported on the low cycle fatigue behavior of refractory fiber/superalloy composites. The low cycle fatigue behavior for tungsten fiber reinforced nickel was determined at room temperature in Ref. 38. Specimens containing 11 to 25 volume percent, 500 micrometer (0.020 in.) diameter, tungsten fibers or 20 to 28 volume percent, 100 micro-

meter (0.004 in.) diameter, tungsten fibers were fabricated by a liquid metal infiltration process and tested in fatigue. Specimens containing 8 or 10 volume percent of 300 micrometer (0.012 in.) diameter tungsten fibers were fabricated by a powder and subsequent forging process and also tested in fatigue. Fatigue tests were performed at about 150 Hz using direct stress, tension-tension, axially loaded specimens. Figure 20 is a plot of the ratio of the maximum stress for fatigue failure to ultimate tensile strength for the range of cycles investigated. The observed fatigue ratios shown for the composite specimens were much higher in comparison to some superalloys referenced by the author. The fatigue ratio reported for Nimocast 713C for 10^8 cycles was 0.24 and for Incoloy 901 and Udimet 700 for 10^7 cycles the ratio was 0.14 and 0.17, respectively. As shown in Fig. 20 the fatigue ratio obtained for the composites was greater than 0.65 at 10^6 cycles.

Low cycle fatigue tests were conducted at 760° and 980° C (1400° and 1800° F) on 20 and 35 volume percent W- 1 percent ThO_2 fiber reinforced FeCrAlY composites, Ref. 36. Fatigue tests were performed at 0.65 Hz and a stress cycle of from 5.5 MN/m^2 (0.8 ksi) to a maximum stress. The load ratio, R, was approximately 0.01 and the stress ratio, A, was 1. The low cycle fatigue results are plotted in Fig. 21. The results indicate that the fiber controls low cycle fatigue strength as was the case for high cycle fatigue behavior. The 35 volume percent fiber content specimens had much higher values of fatigue strength versus cycles to failure than did the 20 volume percent fiber specimens. The ratio of fatigue strength to ultimate tensile strength versus cycles to failure is plotted in Fig. 22. Very high values were obtained at both 760° and 980° C (1400° and 1800° F) indicating that the composite has a high resistance to low cycle fatigue in this temperature range.

c. Thermal Fatigue

Thermal fatigue failures are caused by the repeated application of stress that is thermal in origin. Rapid changes in the temperature of the environment can cause transient temperature gradients in turbine engine components. Such temperature gradients give rise to thermal stresses and strains. Thermal fatigue failure is the cracking of materials caused by repeated rapid temperature changes.

Superimposed on stresses generated by temperature gradients, in the case of the composite, are internal stresses caused by the difference in expansion coefficients between the fibers and matrix. The mean coefficient of thermal expansion from room temperature to 1100° C (2010° F) for superalloys ranges from 15.8 to $19.3 \times 10^{-6}/^{\circ}\text{C}$ (8.8 to $10.7 \times 10^{-6}/^{\circ}\text{F}$) and is approximately $5 \times 10^{-6}/^{\circ}\text{C}$ ($2.7 \times 10^{-6}/^{\circ}\text{F}$) for tungsten. Because of the large difference in expansion coefficients between the fiber and matrix and the resulting strains, thermal fatigue is believed to be the most serious limitation on composite usefulness.

A number of investigators have developed analytical methods to calculate the dependence of composite deformation on cyclic, geometric and constituent deformation parameters, Refs. 39 to 41. The results of these calculations illustrate the possible effects of several variables on deformation damage parameters. Because of the difference in expansion coefficients, the matrix is strained in tension upon cooling and in compression upon heating while the fiber is strained in compression upon cooling and tension upon heating.

Figure 23 is a plot showing the stress on the matrix as a result of heating and cooling. During cooling (A to B) the matrix stress increases continuously in tension because the matrix contracts faster than does the

fiber. After reaching the lowest temperature, point B, and upon reheating the matrix is strained elastically in compression up to point C and the stress falls linearly. At point D the temperature and stress are high enough to allow matrix creep, and yielding of the matrix occurs. Because the temperature continually increases above point D, creep becomes much easier and the stress on the matrix continuously decreases until the original maximum temperature is achieved, point E. If, upon reaching point E, the temperature is held constant the matrix stress would fall further due to matrix creep with time. The magnitude of the stress on the matrix is dependent upon the volume fraction fiber present in the composite. The larger the volume fraction fiber present in the composite the higher the stress on the matrix and the lower the temperature at which the matrix would yield upon heating. Yielding of the matrix could also occur in tension during cooling. Work reported in Ref. 39 indicates that the hysteresis loop of matrix stress versus temperature caused by plastic deformation of the matrix stabilizes after a few cycles so that a steady-state plastic compression-tension fatigue results when no external stress is present. Total cyclic plastic strain increases by a law of the form, total strain = strain per cycle x number of cycles. The ability of the matrix to accommodate plastic strain thus controls the number of cycles to failure for the composite if plastic deformation of the matrix governs the failure mode of the composite in fatigue.

A similar type plot could be constructed for the stress on the fiber as a function of temperature. The stress on the fiber during cooling would be compressive and upon heating the stress would be tension. At low volume fraction fiber contents the compressive stress on the fiber during cooling could be large enough to cause plastic flow of the fiber as indicated in

Ref. 40. The stress on the matrix and fiber is also dependent upon the maximum cycling temperature, heating-cooling rate and creep rate. Strain induced damage increases markedly with increases in the maximum cycling temperature. Decreasing the heating-cooling rate or increasing the creep rate has the same effect. A decrease in heating-cooling rate increases the range of plastic strain per cycle, simultaneously decreasing the maximum matrix stress. Three types of cycling damage have been noted to date: plastic flow of the fiber in compression, matrix fracturing and fiber-matrix interface debonding.

A number of studies have been conducted on the response of tungsten fiber/superalloy composites to thermal cycling. Table V compares the data obtained for several composite systems. Cylindrical specimens of 40 percent W/Nimocast 258 were cycled between room temperature and 1100° C (2010° F) in a fluidized bed to obtain rapid heating and cooling, Ref. 29. Metallographic examination after 400 cycles revealed no apparent damage at the fiber-matrix interface. Presumably no matrix or fiber cracks were observed or they would have been reported as such. Cylindrical specimens of 13 percent W/Nimocast 713C were cycled in a fluidized bed in the temperature ranges shown in Table V, Ref. 16. Cracking occurred after relatively few cycles with the exception of the specimens cycled from 20° to 600° C (70° to 1110° F). The cracking was not extensive for the 550° to 1050° C (1020° to 1920° F) cycle tests, but was extensive in the 20° to 1050° C (70° to 1920° F) cycle tests. The bond between the fiber and matrix was reported to be severely degraded by thermal cycling to 1050° C (1920° F). Thermal cycle tests were conducted on specimens of reinforced sheet material having a matrix of EI435 (Nichrome) and volume fiber contents of 14, 24 or 35 percent, Ref. 42. The specimens were heated in an electric resistance furnace

for 2.5 minutes up to a temperature of 1100° C (2010° F) followed by a water quench to room temperature. The number of cycles for debonding between the fiber and matrix to occur was determined as a function of fiber content. As shown in Table V the number of cycles for debonding to occur decreased with increasing fiber content. The tests also indicated that one of the inherent requirements of the fiber, from the viewpoint of obtaining the best thermal fatigue response, is a strict uniformity of spacing of the fibers throughout the body of the material. Thermal fatigue failure occurred first where fibers were in contact with one another. Concurrently, where fibers were congested, cracks formed in the matrix along surfaces paralleling the fiber axis. Tests were also conducted on reinforced EI435 sheet material in Ref. 43. The fiber contents investigated were 15 or 32 percent. The specimens were heated by passage of an electric current. Specimens were heated and cooled in 30 seconds in the temperature ranges shown in Table V. Irreversible deformation occurred after cycling for all of the 15 volume fiber content specimens but not for the 32 volume fiber content specimens. During the initial stages of cycling, warpage and bending were observed. A length decrease was observed during the entire test. With an increase of the number of cycles the length and rate of the dimensional change diminished. After 1000 cycles from 600° to 1100° C (1110° to 2010° F) the length of the specimen decreased 20 percent. Heat treatment had a considerable effect on dimensional instability of the composite. As a result of annealing the specimen their propensity for deformation during thermocycling decreased. After annealing at 1100° C (2000° F) for 4 hours specimens cycled from 570° to 1000° C (1050° to 1830° F) for 1000 cycles did not change their shape. Annealing reduced the yield strength of the matrix. The level of stresses arising in the fibers as a consequence of the differ-

ence in expansion coefficients was determined by the resistance to plastic deformation of the matrix. With a decrease in the yield strength of the matrix the level of stress on the fiber decreased and the fibers did not plastically deform. Cracks at the interface were observed for specimens containing 15 volume percent fiber while cracks in the matrix between the fibers were observed for specimens containing 32 volume percent fibers. Several different nickel base composite systems were thermally cycled in Ref. 44. Specimens containing 35 or 50 volume percent fiber were heated by passage of an electric current. The specimens were heated to 1093° C (2000° F) in 1 minute and cooled to room temperature in 4 minutes. All of the 35 volume fiber content specimens were warped after 100 cycles, while the 50 volume percent fiber content specimens were not. Two of the nickel base composite materials containing 35 volume percent fiber experienced a decrease in specimen length after 100 cycles. The most ductile matrix materials NiCrAlY showed the least amount of damage after 100 cycles. The 50 volume percent fiber content specimen containing a NiCrAlY matrix was cycled from 427° to 1093° C (800° to 2000° F) for 1000 cycles and experienced internal microcracking in the matrix between the fibers. Specimens containing 30 volume percent W-1 percent ThO_2 fibers in a matrix of FeCrAlY were exposed to 1000 cycles from room temperature to 1204° C (2200° F), Ref. 45. The specimens were heated up to 1204° C (2200° F) in 1 minute and cooled to room temperature in 4 minutes. As shown in Fig. 24, surface roughening occurred, but there was no matrix or fiber cracking after the 1000 cycle exposure.

As indicated in Table V, a composite system has been identified, W-1 ThO_2 /FeCrAlY, that can be thermally cycled though a large number of cycles without any apparent damage. With the exception of the 40 percent

W/Nimocast 258 composite system which withstood 400 cycles without any apparent damage, all of the other systems investigated indicated that some type of damage occurred. These systems would be limited to applications where the component would be exposed to very few thermal cycles. Only a limited number of systems have been investigated to date and a need exists to identify other thermal fatigue resistant systems. The results obtained indicate that a ductile matrix which can relieve thermally induced strains by plastic deformation is required for composite thermal fatigue resistance.

Impact Strength

Composite materials must be capable of resisting impact failure from foreign objects or from failed components that may pass through the engine if they are to be considered for use as a blade or vane.

Factors affecting the impact strength of tungsten fiber metal matrix composites were investigated in Ref. 46. Miniature Izod and standard Charpy impact strength data were obtained for a tungsten fiber reinforced nickel base alloy (Ni-25W-15Cr-2Al-2Ti). It has been found that composite properties as measured by the miniature Izod impact test correlate closely with composite properties as measured by various ballistic impact tests and it was concluded that the miniature Izod test is a reasonable screening test for candidate turbine blade and vane materials, Ref. 47. The Izod impact strength of unnotched and notched specimens as a function of fiber content is plotted in Fig. 25 for two test temperatures, (75° and 1000° F). Impact strength decreased with increasing fiber content at the lower temperature, but increased with increasing fiber content at the higher temperature.

Figure 26 is a plot of impact strength as a function of temperatures. There is a sharp increase in impact strength for the 60 volume percent unnotched specimen at 260° C (500° F), the ductile-brittle transition temperature

(DBTT) for the fiber. In general unnotched composites had higher impact strength compared to the matrix at temperatures above the DBTT of the fiber and lower impact strength than the matrix below the DBTT of the fiber. The matrix's contribution to impact strength for the composite is most significant at low temperatures while the fiber controls higher temperature impact strength, above 260° C (500° F). The effect of fiber content on notch sensitivity was also determined. The ratio of the composite's notched impact strength per unit area to its unnotched impact strength per unit area is plotted as a function of fiber content in Fig. 27. The notch sensitivity of the composite decreased with increasing fiber content both above and below the DBTT of the fiber. Heat treatment or hot rolling improved the room temperature impact strength of the composite. Heat treatment increased the impact strength of the notched unreinforced matrix by almost four times and nearly doubled the impact strength of a 45 volume percent fiber content composite. Round rolling increased the impact strength of a 56 volume percent fiber content composite by nearly four times. The improved impact strength for the composite was related to improved matrix impact strength.

An additional objective of work conducted in Ref. 46 was to determine if the potential impact resistance of tungsten fiber/superalloy composites was sufficient to warrant their consideration as turbine blade or vane materials. Alloys with miniature Izod impact values less than 1.7 joules (15 in.-lb) have been successfully run as turbine blades (Refs. 48-49). Based on this, the value of 1.7 joules (15 in.-lb) was taken as the minimum value for Izod impact strength to indicate if a material has promise for further evaluation leading to turbine blade use. Figure 28 compares the tungsten fiber/superalloy impact strength values with the minimum standard. At room temperature as fabricated composites containing fiber contents

greater than 35 percent did not meet the minimum requirement. Heat treatment and hot working, however, improved the impact strength so that high fiber content composites met the minimum requirement. At 760° C (1400° F), the higher fiber content as-fabricated composites have impact strengths distinctly above the minimum requirement. High Charpy impact strength values were obtained at 1093° C (2000° F), 37.3 J (330 in.-lb) for a 60 volume percent fiber content specimen, implying that most of this strength is maintained to at least 1093° C (2000° F). The impact strength potential for tungsten fiber/superalloy composites thus appears adequate for turbine blade and vane applications.

A further comparison of the room temperature and 760° C (1400° F) miniature Izod impact strength for some superalloys and other composite systems is plotted in Fig. 29. Values for the Inconel-713C, 25 percent W/Nichrome and WI-52 were obtained from Ref. 26 while data for the 25 percent W-1 percent ThO₂/Hastelloy X are from Ref. 47. Inconel-713C and Guy Alloy represent past turbine blade materials while the WI-52 alloy is representative of an older vane material. The room temperature impact strength values obtained for the composites all exceed the minimum standard, which is equal to the value for Guy Alloy, as well as that for the vane material WI-52. At 760° C (1400° F) the 25 percent W/Nichrome composite bent but did not fracture when impacted at 127 J (157 in.lb) at a velocity of 54 cm per second (136 in. per second). At 760° C (1400° F) the 25 percent W-1 percent ThO₂/Hastelloy X composites bent and cracked, but the crack did not extend or completely propagate through the cross section of the specimen. The impact strength of the composites were thus much higher than that obtained for Inconel 713C or WI-52 at 760° C (1400° F) and should be adequate for turbine blade or vane applications.

Oxidation and Corrosion

The gaseous environment in the gas turbine engine is highly oxidizing with oxygen partial pressures of the order of 2-4 atm, Ref. 50. However, this environment also contains significant amounts of combustion product impurities including sulfur from the fuel and alkali salts ingested with the intake air. Under these conditions an accelerated oxidation may be encountered, sometimes, but not always, accompanied by the formation of sulfides within the alloy: this is commonly referred to as hot corrosion.

The basic design of the composite material assumes that the superalloy matrix will provide oxidation resistance, including protection of the tungsten fibers. Superalloys that are used for hot section engine components are oxidation resistant for material operating temperatures up to about 980° C (1800° F). Above a material temperature of 980° C (1800° F) it is necessary to coat or clad the material to provide the required oxidation resistance. Claddings that are used for superalloy oxidation protection such as NiCrAlY and FeCrAlY are oxidation resistant to temperatures above 1090° C (2000° F). These materials may also be considered as the matrix for composites so that the composite would not have to be coated or clad for high temperature oxidation resistance. Preliminary oxidation studies were conducted in Ref. 27 on a nickel base superalloy reinforced with tungsten-1 percent ThO_2 fibers and clad with Inconel. The specimens were exposed in air at 1100° C (2010° F) for times up to 300 hours. Figure 30 is a transverse section of a clad composite specimen exposed for 50 hours at 1093° C (2000° F). The Inconel cladding was oxidized, and a coherent oxide scale formed on the Inconel. Oxidation had not progressed to the composite, and the surface fibers were not affected by the oxidation of the cladding. Composite specimens of W-1 percent $\text{ThO}_2/\text{FeCrAlY}$ having completely matrix

protected fibers were exposed to static air at 1038°, 1093° and 1149° C (1900°, 2000°, and 2100° F) for up to 1000 hours. The weight change in 1000 hours was 0.3 mg/cm² for 1038° C (1900° F) and 1.26 mg/cm² for 1149° C (2100° F). These values are in agreement with values obtained for the matrix material without any reinforcement, Ref. 51. Oxidation did not progress to the surface fibers.

The oxidation and corrosion resistance of composite materials having exposed fibers is also an important consideration. Although the fibers in the composite would not be designed to be exposed to the engine environment an understanding of the high temperature oxidation and corrosion behavior is desirable in the event of a coating, cladding, or matrix failure during service which could occur, for example, from impact due to foreign objects passing through the engine. Figure 31 illustrates the principal paths for oxidation and corrosion of exposed fibers. Oxidation proceeding perpendicular to the fibers (through the blade or vane thickness) would destroy the exposed fibers, but intervening matrix would prevent oxidation of subsequent layers. Thus only a partial loss of strength would result. Oxidation parallel to the fibers (along the blade or vane span) potentially is more severe, since all the exposed fibers in the cross section potentially could be oxidized along their entire length. However, studies conducted to evaluate oxidation along fibers showed only limited oxidation penetration along the fibers, Refs. 45, 50, and 52. In Ref. 45 it was found that after 10 hours exposure to static air at 1200° C (2190° F), the fibers in W-1 percent ThO₂/FeCrAlY were oxidized to a depth of only 2.5 mm (0.1 in.). After 100 hours exposure at 1100° C (2010° F), the fibers in a W/Nimocast 258 composite oxidized to a depth of 1.3 mm (0.05 in.) in static air and to 2.5 mm (0.1 in.) in a low velocity simulated engine exhaust gas stream moving at

1.8 m/s (6 ft/s), Ref. 52. Measurements of the depth of fiber oxidation for end exposed tungsten fibers in oxidized tungsten fiber reinforced Ni-20 percent Cr specimens obtained from photographs presented in Ref. 50 indicate similar values. In Ref. 50, however, considerable distortion and degradation of the matrix surrounding the oxidized tungsten fiber was observed. Typical weight gain-time curves for the oxidation of 40 volume percent tungsten fiber content nickel- 20 percent chromium material tested at 900° and 1000° C (1650° and 1830° F) in 1 atmosphere oxygen are shown in Fig. 32, Ref. 50. While tungsten can form volatile oxides, the large weight gains reflected in these data indicate the possible formation of complex matrix-tungsten oxides.

The hot corrosion behavior of tungsten fiber reinforced Ni-20 percent Cr composite specimens was also examined in Ref. 50, under the following exposure conditions: (a) sulfidation in H₂-10 percent H₂S; (b) presulfidation in H₂-10 percent H₂S followed by oxidation in oxygen; and (c) oxidation in 1 atmosphere of oxygen after precoating with Na₂SO₄. During sulfidation, only the matrix formed sulfides and the fibers remained unaffected. Consequently, presulfidation, although having a dramatic effect on the oxidation of the matrix did not have a damaging effect on the fibers. The presence of sodium sulfate was also not critical. Thus, hot corrosion conditions were not harmful to the tungsten reinforced composites studied, and catastrophic loss of the exposed tungsten fibers did not occur upon exposure to a high-temperature oxidizing environment.

Thermal Conductivity

High thermal conductivity is desirable in a turbine blade material to reduce temperature gradients; this, in turn, results in reduced thermally induced strains that can cause cracks or distortion. In addition, higher

thermal conductivity can reduce coolant flow requirements in some impingement cooled blades leading to greater engine efficiency or durability (Ref. 53). High conductivity for some turbine blade applications could result in unacceptable higher disc temperatures. However, proper design could alleviate the problem, Ref. 54. The conductivity of tungsten fiber/superalloy composites is markedly superior to that of superalloys. The thermal conductivity of tungsten is much higher than that for superalloys and the more tungsten added to a composite the greater the conductivity. Thermal conductivity of the composite is greatest in the direction of the fiber axes since there is a continuous path for conduction along the tungsten fibers. Conduction perpendicular to the fiber axes is lower because the heat cannot find a continuous path through tungsten. The thermal conductivity of some tungsten fiber/superalloy composites was determined as a function of temperature in Ref. 53. Figure 33 shows the thermal conductivity values obtained for a composite containing 65 volume percent fibers in a nickel base alloy and tested in the direction of the fiber axis and for a composite containing 50 volume percent fiber and tested in the direction perpendicular to the fiber axis. The longitudinal thermal conductivity is seen to be much higher than the transverse conductivity. Also shown in the figure are values for the matrix materials. The composites have much higher values for thermal conductivity over the entire temperature range. The results reported in Ref. 53 indicate that at blade conditions (30 to 60 volume percent fiber content and 730° C to 1130° C (1350° to 2060° F)) the transverse (through the wall) conductivity of the composite will be 35 to 50 W/mK. The longitudinal (spanwise) conductivity will be about 45 to 65 W/m-K. Typical superalloys have conductivities of about 25 W/m-K at 1030° C (1880° F).

Composite Turbine Blade Material Requirements

Relatively small increases in turbine component use temperature such as 6° C (10° F) are beneficial and result in cost savings in aircraft engine operation. Numerous modifications in engines, including material changes, have been made to further such gains. However, the introduction of a fiber reinforced superalloy component, such as a tungsten fiber reinforced superalloy turbine blade would represent a large material change. Such a change would require a larger potential gain to justify the considerable effort required to make the application possible.

It has been assumed in the program at NASA-Lewis that a composition selected as a serious turbine component material candidate must achieve at least a 50° C (90° F) use temperature advantage over the best currently used superalloys and the goal of our program is to permit a 100° to 150° C (180° to 270° F) use temperature increase.

The properties of the composite are controlled by the fiber and matrix properties. Therefore, the properties needed in the composite dictate the fiber and matrix materials which may be used. Hence, selecting the composite involves selecting the best compromise in the combination of fiber and matrix alloys used. Specific composite fiber and matrix requirements are as follows:

(a) Fiber Property Requirements

Creep-rupture and mechanical fatigue strength must be adequate to permit at least a 50° C (90° F) composite blade metal temperature advantage over current superalloys.

Toughness must be adequate at operating temperatures to insure the needed foreign object damage (FOD) resistance.

Fiber costs must be low so that blade fabrication costs can be kept acceptably low.

(b) Matrix Property Requirements

Compatibility with the fibers is required at fabrication and operating temperatures so that fiber strength is not excessively degraded by interdiffusion.

Mechanical and thermal fatigue resistance is needed at operating temperatures. Thermal fatigue damage in the matrix can be initiated by the thermal expansion coefficient mismatch between fiber and matrix as well as by large temperature gradients.

Oxidation and hot corrosion resistance are needed in the matrix for use at temperatures up to 1100° C (2010° F). Not only is this a severe temperature in general, but at these levels the fibers lack oxidation resistance and must be protected by the matrix.

Density must be low to help offset the high density of the tungsten fibers. This is a particularly important consideration in aircraft engines where weight must be minimized because high blade densities can lead to high disk weights.

Toughness and ductility must be high at low temperatures because the matrix imparts impact damage resistance to the composite at low temperatures.

Shear creep strength must be adequate to allow fiber angle plying for airfoil chordwise strength and torsional strength.

First Generation Composite Turbine Blade Material Selection

Tungsten-fiber/FeCrAlY was identified as a promising first generation turbine blade material because of the excellent combination of complementary properties possible with this combination of fiber and matrix, Ref. 55. The matrix provides a high melting point, low density, excellent oxidation and hot corrosion resistance, limited fiber-matrix interdiffusion at proposed blade temperatures, as well as excellent ductility to aid in thermal fatigue

resistance. The fiber provides high stress-rupture, creep, fatigue, and impact strength along with high thermal conductivity. Properties reported for this material indicate that it has adequate properties for turbine blade use and it could permit turbine blade operating temperatures over 50° C (90° F) greater than those of current directionally solidified (DS) super-alloy blades.

Composite Component Fabrication

Having demonstrated adequate properties for application as a turbine blade material, the next area of consideration is whether complex shapes such as hollow turbine blades can be designed and fabricated from such material and at reasonable cost. The selected composite fabrication techniques must result in a composite whose properties meet those required for application of the composite. The processes must be capable of first producing component shapes to required dimensions, second incorporating both uniaxial and off-axis fiber positioning; third providing uniform matrix cladding to prevent fiber oxidation, and fourth, providing if necessary, for cooling or weight reduction passages. The combined fabrication techniques must also be cost effective and reproducible.

Investment casting has been considered for fabrication of composite blades. However, there are two obstacles to overcome. A way must be found to hold the fibers in positive "angle plied" alignment during infiltration by the molten matrix. Also, small diameter fibers which must be used in hollow blades because of a space limitation are subject to damage caused by fiber-matrix interdiffusion and to displacement during casting. A W-2 percent ThO_2 /MAR M322 composite JT9D turbine blade was fabricated by a casting technique reported in Ref. 19. Separation of the fiber bundle from the matrix occurred in the concave areas of the blade. Test specimens fabri-

cated by the same process revealed that outer fibers are exposed to higher temperatures than inner fibers. Furthermore, the matrix composition became richer in tungsten which indicated dissolution of the fibers. Given the current state of the art, investment casting would appear to be more suitable for fabrication of uniaxially reinforced solid blades in which large fibers can be used.

Diffusion bonding of monolayer composite plies is currently the most promising, cost effective method of fabrication for a hollow blade. The composite plies consist of aligned tungsten fibers sandwiched between layers of matrix material. This approach has the capability for accurate fiber distribution and alignment; moreover, it limits fiber-matrix interdiffusion during fabrication. This approach also is capable of producing blade shapes that are close to final dimensions; hence only root machining and touch up grinding is needed. Solid B/Al and B/Ti fan and compressor blades have been fabricated using this approach. A solid W-2 percent ThO_2 /conventionally cast MAR-M200 prototype airfoil for potential application in an advanced industrial gas turbine engine was also fabricated, Ref. 56, using a similar approach.

The feasibility of fabricating a composite hollow turbine blade was successfully demonstrated in work reported in Ref. 57. A JT9D-7F first stage, convection cooled blade was selected as the model from which a W-1 percent ThO_2 /FeCrAlY composite blade was designed. The major purpose of the fabrication effort was not only to demonstrate the feasibility of fabricating a hollow blade but to also demonstrate that design requirements could be met in the fabricated blade. The design features incorporated into the fabricated blade are indicated in Figs. 34 and 35. The external airfoil was identical to that of the current MAR-M200 (D.S.), JT9D-7F blade. However,

the airfoil walls were designed to be thinner to reduce the composite blade weight to within 10 percent of the current blade weight and to allow for more efficient air cooling. An impingement cooling insert was also added to improve cooling efficiency. The walls of the airfoil were built up of composite plies containing W-1 percent ThO_2 fibers. The inner and outer plies of the wall consisted of unreinforced FeCrAlY for oxidation resistance. The composite airfoil extends down into the blade root. The matrix used in the airfoil above the root was FeCrAlY for oxidation and thermal fatigue resistance. The matrix used in the airfoil within the root was an alloy optimized for shear strength and thermal fatigue resistance. The root requires high strength superalloy or a composite built-up of plies graded for differing thermal expansion to minimize thermal fatigue problems at the airfoil-root interface. The blade was designed to have a potential 50° C (90° F) use temperature advantage over the current MAR-M-200 (D.S.) blade.

Figure 36 shows the fabrication sequence used to produce the hollow composite turbine blade. A tungsten fiber mat was sandwiched between powder sheets of FeCrAlY which was subsequently hot pressed to form a monotape. The monotape was then cut into the plies necessary to arrive at the final blade dimension. The plies were then stacked around a steel core. Root inserts and outserts could also be stacked around the assembly or could be attached in a secondary fabrication step. The entire assembly was then placed in a refractory metal die, heated and pressed to arrive at the proper airfoil contour. After pressing the steel core was removed by leaching out with an acid. A tip cap was then welded on to the end of the airfoil and an impingement cooling insert was placed in the leached out cavity and brazed to the root of the blade.

Figure 37 shows the as fabricated W-1 percent $\text{ThO}_2/\text{FeCrAlY}$ composite hollow JT9D-7F airfoil containing a bonded on end cap and trailing-edge coolant slots. Figure 38 shows the composite airfoil which was brazed to a high strength superalloy arc root. A cross section of the composite airfoil is shown in Fig. 39. Excellent fiber alignment and fiber distribution was obtained and the fibers were fully protected by a layer of FeCrAlY in the interior and exterior of the airfoil.

Successful fabrication of a hollow composite airfoil has demonstrated that this material can be fabricated into the complex design shapes for hot turbine section components. While components such as vanes or combustion liners have not been fabricated these components are less complex than the blade and can be considered for future programs.

The fabrication process sequence used to produce a hollow composite blade was used in a fabrication cost study (Ref. 58). Fabrication costs were estimated for high technology turbine blades fabricated using three different materials. The same turbine blade configuration, a first stage JT9D-7F blade was used for each material. Directionally solidified eutectic (DSE), an oxide dispersion strengthened superalloy (ODSS), and W-1 percent $\text{ThO}_2/\text{FeCrAlY}$ blade manufacturing costs were compared with the cost of producing the same blade from a DS superalloy, the current blade material. The relative costs are shown in Fig. 40. The study indicates that $\text{W}/\text{FeCrAlY}$ manufacturing costs should be competitive with current manufacturing costs of manufacturing this blade by directional solidification of a superalloy - provided the projected manufacturing yields can be realized in actual commercial production of blades.

CONCLUDING REMARKS

Exploratory development and material property screening have indicated that tungsten fiber reinforced superalloy composites have considerable potential for application as advanced high temperature turbine engine component materials. A first generation TFRS composite has been selected to serve as a demonstration system to evaluate the merit and problems of this family of composites for turbine engine applications. Based on the data obtained from the development and property screening, thoriated tungsten wire in a FeCrAlY matrix has potential to become a viable candidate for application.

The choice of commercially available thoriated tungsten wire was made despite the appreciable difference in properties obtainable when stronger wire such as W-Hf-C were used because of current cost and availability. The manufacturing technology effort to make the stronger wire available can more readily be justified after TFRS has become better accepted for application. Test parameters can be evaluated equally well with the less expensive, readily available wire.

The choice of FeCrAlY as a matrix is based on the ductility, low strain hardening and oxidation and corrosion resistance of the alloy and good chemical compatibility with tungsten fibers. The oxidation-corrosion resistance of the composite can be achieved by a thin layer on the external and internal surfaces of a component. The ductility and low strain hardening rate of the matrix are vital to resist the plastic deformation that occurs from thermally induced strains from thermal gradient and thermal expansion mismatch between the fiber and matrix. FeCrAlY is not the ideal matrix because its thermal expansion coefficient is much higher than the fibers and its shear strength is low at elevated temperatures. As in the

case of the thoriated tungsten wire, however, it is a reasonable choice as a first generation system to indicate that TFRS is a viable material for further improvement.

Use of tungsten/FeCrAlY composite material could permit engine turbine blade temperatures over 50° C (90° F) higher than those possible using conventional superalloys. Moreover, blade fabrication studies have demonstrated the feasibility of producing hollow W/FeCrAlY turbine blades at a cost competitive with DS superalloy blade costs. Still, a great deal of work remains to be done on this material to aid in its transition from laboratory feasibility to rig testing of prototype hardware and then on manufacturing technology and detailed design. Current Lewis Research Center efforts are addressing these problems.

REFERENCES

1. L. H. Amra, L. F. Chamberlin, F. R. Adams, J. G. Tavernelli and G. J. Polanka, "Development of Fabrication Process for Metallic Fibers of Refractory Metal Alloys," NASA CR-72654, 1970.
2. G. W. King, "Development of Wire-Drawing Processes for Refractory-Metal Fibers," NASA CR-120925, 1972.
3. D. W. Petrasek and R. A. Signorelli, "Stress-Rupture and Tensile Properties of Refractory-Metal Wires at 2000° and 2200° F (1093° and 204° C)," NASA TN D-5139, 1969.
4. D. W. Petrasek, "High-Temperature Strength of Refractory-Metal Wires and Consideration for Composite Applications," NASA TN D-6881, 1972.

5. D. W. Petrasek and J. W. Weeton, "Alloying Effect on Tensile Properties and Micro-Structure of Tungsten-Fiber-Reinforced Composites," NASA TN D-1568, 1963.
6. T. Montebano, J. Brett, L. Castleman and L. Seigle, "Nickel Induced Recrystallization of Doped Tungsten," Trans. Metall. Soc. AIME, 242, 1973-1979 (1968).
7. J. Hoffman, S. Hofmann and L. Tillmann, "Recrystallization of Tungsten Fibers in Nickel Matrix Composites," Z. Metallk., 65, 721-726 (1974).
8. H. Gruenling and G. Hofer, "Deferred Recrystallization of Tungsten Wire in Nickel and Nickel-Chromium Matrices," Z. Werkstofftech., 5 (2), 69-72 (1974).
9. D. W. Petrasek, R. A. Signorelli and J. W. Weeton, "Refractory-Metal-Fiber-Nickel-Base-Alloy Composites for Use at High Temperatures," NASA TN D-4787, 1968.
10. D. M. Karpinos, L. I. Tuchinskii, I. R. Vishnyakov, L. N. Pereselentseva and L. N. Klimenko, "Effect of Alloying a Nickel Matrix With The Metal Of Reinforcing Fibers On Structural Stability Of Nickel-Tungsten and Nickel-Molybdenum Composite Materials," Fiz. Khim. Obrab. Mater., 6, 107-113 (1972).
11. B. A. Klypin, A. M. Maslov and S. B. Maslenkov, "Effect of Alloying of the Structural Stability of Ni-W Co-W Composite Materials," Metalloved. Term. Obrab. Met., 5, 6-11 (1977). Transl. Met. Sci. Heat Treat. Met., 19 (5-6) 343-348 (1977).
12. V. S. Mirotvorskii and A. A. Ol'shevskii, "Reactions of Tungsten Fibers With Cobalt-Base Matrices," Poroshk. Metall., 7 (187), 57-64 (1978). Transl. Sov. Powder Metall. Met Ceram., 17 (7), 536-541 (1978).

13. V. S. Mirotvorskii and A. A. Ol'shevskii, "Reactions of Thoriated Tungsten Fibers with Iron-Base Powder Matrices," Poroshk. Metall., 7 (163), 46-52 (1976). Transl. Sov. Powder Metall. Met. Ceram., 15 (7), 534-540 (1976).
14. W. D. Brentnall, "Metal Matrix Composites for High Temperature Turbine Blades," TRW-ER-7790-F, TRW, Inc., Cleveland, Ohio, 1976 and NADC-76225-30, Naval Air Development Center, Warminster, Penn., 1976.
15. V. S. Mirotvorskii and A. A. Ol'shevskii, "Interaction of Thoriated Tungsten At 1200°-1600° C With Matrices Based On Various Metals," Metalloved. Term. Obrab. Met., 11, 12-15 (1979). Transl. Met Sci. Heat Treat. Met., 21 (11-12), 826-829 (1980).
16. A. W. H. Morris and A. Burwood-Smith, "Fiber Strengthened Nickel-Base Alloy," High Temperature Turbines, AGARD-CP-73-71, Jan. 1971.
17. R. A. Signorelli, "Review of Status and Potential of Tungsten Wire: Superalloy Composites for Advanced Gas Turbine Engine Blades," NASA TM X-2599, 1972.
18. R. J. E. Glenny, "Fibrous Composites with High Melting-Point Matrices," Proc. Roy. Soc., Ser. A, 319 (1536), 33-44 (1970).
19. I. Ahmad and J. M. Barranco, "Reinforced Cobalt Alloy Composite For Turbine Blade Application," SAMPE Q., 8, 38-49 (1977).
20. D. M. Karpinos, L. I. Tuchinskii, L. R. Vishnyakov and V. Ya. Fefer, "Expenditure of Energy in the Free Forging of Reinforced Metal Composites," Poroshk. Metall., 6 (150), 20-26 (1975). Transl. Sov. Powder Metall. Met. Ceram., 14 (6), 447-452 (1975).

21. V. P. Severdenko, A. S. Matusevich and A. E. Piskarev, "Production of Composite Sheet Materials Based On KhN77TYuR and VZh98 Alloys," Poroshk. Metall., 6 (138), 51-54, (1974). Transl. Sov. Powder Metall. Met. Ceram., 13 (6), 476-479 (1974).
22. W. D. Brentnall and I. J. Toth, "Fabrication of Tungsten Wire Reinforced Nickel-Base Alloy Composites. NASA CR-134664, 1974.
23. P. R. Sahm, "Eutectic and Artificial Composite Superalloys," pp. 73-114 in Proceedings of the Third Symposium On High-Temperature Materials In Gas Turbines, Edited by P. R. Sahm, Elsevier Scientific Publishing Co., Amsterdam, 1974.
24. R. J. E. Glenny and B. E. Hopkins, "Gas Turbine Requirements," Philos. Trans., R. Soc. (London), Series A, 282 (1307), 105-118 (1976).
25. W. Endres, "Design Principles of Gas Turbines," in pp. 1-14 in Proceedings of the Third Symposium on High-Temperature Materials in Gas Turbines, Edited by P. R. Sahm, Elsevier Scientific Publishing Co., Amsterdam, 1974.
26. A. R. Stetson, B. Ohnysty, R. J. Akins and W. A. Compton, "Evaluation Of Composite Materials For Gas Turbine Engines," AFML-TR-66-156, Part 1, Air Force Materials Lab., Wright-Patterson AFB, OH, June 28, 1966.
27. D. W. Petrasek and R. A. Signorelli, "Preliminary Evaluation of Tungsten-Alloy-Fiber-Nickel-Base Alloy Composites for Turbojet Engine Applications," NASA TN D-5575, 1970.
28. D. W. Petrasek and R. A. Signorelli, "Stress-Rupture Strength and Micro-structural Stability of Tungsten-Hafnium-Carbon-Wire Reinforced Superalloy Composites," NASA TN D-7773, 1974.

29. A. V. Dean, "The Reinforcement of Nickel-Base Alloys with High-Strength Tungsten Wires," J. Inst. Met., 95, 79-86 (1967).
30. V. M. Chubarov, Yu V. Levinskii, S. E. Salibekov, A. F. Trefilov, L. V. Grachev, E. M. Rodin, M. Kh. Levinskaya and L. V. Dvoichenkova, "A Nickel Base Heat Resistant Composite Material," Probl. Prochn., 3, (7), 100-104 (1971). Transl. Strength Mater., 3 (7), 856-859 (1972).
31. A. W. H. Morris and A. Burwood-Smith, "Some Properties of a Fiber-Reinforced Nickel-Base Alloy," Fibre Sci. Technol., 3 (1), 53-78 (1970).
32. G. I. Friedman and J. N. Fleck, "Tungsten Wire-Reinforced Superalloys For 1093° C (2000° F) Turbine Blade Applications," NASA CR-159720, 1979.
33. R. H. Baskey, "Fiber-Reinforced Metallic Composite Materials," AFML-TR-67-196, Air Force Materials Lab., Wright Patterson AFB, OH, 1967.
34. A. Kannappan and H. F. Fischmeister, "High Temperature Stability of Tungsten Fiber Reinforced Nickel Composites," pp. 85-98 in Proceedings of the Fourth Nordic Symposium on High Temperature Materials Phenomena, Vol. II, Physical Metallurgy, Edited by M. Tilli, Helsinki University of Technology, Esbo, 1975.
35. M. O. Speidel, "Fatigue Crack Growth at High Temperatures," pp. 207-255 in Proceedings of the Third Symposium On High-Temperature Materials In Gas Turbines," Edited by P. R. Sahm, Elsevier Scientific Publishing Co., Amsterdam, 1974.
36. J. N. Fleck, "Fabrication of Tungsten-Wire/FeCrAlY - Matrix Composites Specimens," TRW, Inc., Cleveland, OH. TRW Report ER-8076, 1979.

37. C. T. Sims and W. C. Hagel, "The Superalloys. Wiley-Interscience, New York, 1972.

38. N. Nilsen and J. H. Sovik, "Fatigue Of Tungsten Fiber Reinforced Nickel," pp. B51-B54 in Practical Metallic Composites; Proceedings of the Spring Meeting, Institution of Metallurgists, London, 1974.

39. G. Garmong, "Elastic-Plastic Analysis of Deformation Induced By Thermal Stress in Eutectic Composites," Metall. Trans., 5, 2183-2205 (1974).

40. A. A. Baranov and E. V. Yakovleva, "Deformation Of a Composite Material During Thermocycling, II," Probl. Prochn., 8, 50-53 (1975). Transl. Strength Mater., 7 (8), 966-969 (1976).

41. V. V. Gaiduk, A. S. Lavrenko and Yu V. Sukhanov, "Dilatometric Method for Determining Thermal Stresses In Tungsten-Nichrome Composites," Probl. Prochn., 9, 108-111 (1972).

42. G. I. Dudnik, F. P. Banas and B. V. Aleksandrov, "Nature of Failure Of Reinforced Sheets Subjected To Thermal Cycling," Probl. Prochn., 5, 99-100 (1973). Transl. Strength Mater., 5 (1), 106-107 (1973).

43. F. P. Banas, A. A. Baranov and E. V. Yakovleva, "Deformation Of Composite Material During Alternate Heating and Cooling," Probl. Prochn., 6, 82-86 (1975). Transl. Strength Mater., 7 (6), 744-748 (1976).

44. W. D. Brentnall and D. J. Moracz, "Tungsten Wire-Nickel Base Alloy Composite Development," TRW, Inc., Cleveland, OH, TRW ER-7849, NASA CR-135021, 1976.

45. W. D. Brentnall and D. J. Moracz, "Tungsten Wire-Nickel Base Alloy Composite Development," TRW, Inc., Cleveland, OH, TRW ER-7849, NASA CR-135021, 1976.

46. E. A. Winsa and D. W. Petrasek, "Factors Affecting Miniature Izod Impact Strength Of Tungsten-Fiber-Metal-Matrix Composites," NASA TN D-7393, 1973.

47. B. Ohnysty and A. R. Stetson, "Evaluation of Composite Materials For Gas Turbine Engines," AFML-TR-66-156, Part 11, Dec. 1967.

48. R. A. Signorelli, J. R. Johnston and J. W. Weeton, "Preliminary Investigation of Guy Alloy as a Turbojet-Engine Bucket Material for Use at 1650° F," NASA RM E56119, 1956.

49. W. J. Waters, R. A. Signorelli and J. R. Johnston, "Performance of Two Boron-Modified S-816 Alloys in a Turbojet Engine Operated at 1650° F. NASA Memo 3-3-59E, 1959.

50. M. E. El-Dahshan, D. P. Whittle and J. Stringer, "The Oxidation and Hot Corrosion Behavior of Tungsten-Fiber Reinforced Composites," Oxid. Met., 9, 45-67 (1975).

51. C. S. Wukusick, "The Physical Metallurgy and Oxidation Behavior of Fe-Cr-Al-Y Alloys," GEMP-414, General Electric Co., Cincinnati, OH, 1966.

52. A. V. Dean, "The Reinforcement of Nickel-Base Alloys with High Strength Tungsten Wires," NGTE-R-266, National Gas Turbine Establishment, Pyestock, England, 1965.

53. E. A. Winsa, L. J. Westfall and D. W. Petrasek, "Predicted Inlet Gas Temperatures For Tungsten Fiber Reinforced Superalloy Turbine Blades," NASA TM-73842, 1978.

54. H. J. Gladden, "Air Cooling of Disk of a Solid Integrally Cast Turbine Rotor for an Automotive Gas Turbine," NASA TM X-3471, 1977.

55. D. W. Petrasek, E. A. Winsa, L. J. Westfall and R. A. Signorelli, "Tungsten Fiber Reinforced FeCrAlY-A First Generation Composite Turbine Blade Material," NASA TM-79094, 1979.
56. P. J. Mazzei, G. Vandrunen and M. J. Hakim, "Powder Fabrication of Fibre-Reinforced Superalloy Turbine Blades," pp. SC 7.1-SC 7.16 in AGARD Conference Proceedings No. 200 on Advanced Fabrication Techniques in Powder Metallurgy and Their Economic Implications, Advisory Group for Aerospace Research and Development, Paris, France, 1976.
57. P. Melnyk and J. N. Fleck, "Tungsten Wire/FeCrAlY Matrix Turbine Blade Fabrication Study," NASA CR-159788, 1979.
58. C. F. Barth, D. W. Blake and T. S. Stelson, "Cost Analysis of Advanced Turbine Blade Manufacturing Processes," NASA CR-135203, 1977.

TABLE I. - CHEMICAL COMPOSITION OF WIRE MATERIALS (REFS. 3 AND 4)

Material	Weight percent of component								
	W	Ta	Mo	Nb	Re	Ti	Zr	Hf	ThO ₂
Tungsten alloys									
218CS	99.9	—	—	—	—	—	—	—	—
W-1ThO ₂	bal	—	—	—	—	—	—	—	0.95
W-2ThO ₂	—	—	—	—	2.79	—	—	—	1.6
W-3Re	—	—	—	—	4.89	—	—	—	—
W-5Re-2ThO ₂	—	—	—	—	22.54	—	—	—	1.78
W-24Re-2ThO ₂	—	—	—	—	4.1	—	—	—	1.7
W-Hf-C	—	—	—	—	—	—	0.37	—	0.030
W-Re-Hf-C	—	—	—	—	—	—	.38	—	.021
Tantalum alloys									
ASTAR 811C	8.2	bal	—	—	—	1.13	—	.91	—
Molybdenum alloys									
TZM	—	—	bal	—	—	.45	0.085	—	.031
TZC	—	—	bal	—	—	1.18	.27	—	.12
Niobium alloys									
FS85	10.44	27.95	—	bal	—	—	.85	—	.031
AS30	20	—	—	bal	—	—	1	—	—
B88	28.3	—	—	bal	—	—	—	1.94	.58

TABLE II. - REPRESENTATIVE PROPERTIES OF REFRACtORY-ALLOY WIRES (REFS. 3 AND 4)

Alloys	Density, gm/cm ³	Wire diameter, mm	Ultimate tensile strength		Stress for 100-hr rupture		Stress/density for 100-hr rupture, cmx10 ³
			ksi	MN/m ²	ksi	MN/m ²	
A. 1093° C (2000° F) Data							
Tungsten alloys							
218CS	19.1	0.20	126	869	63	434	234
W-1ThO ₂	19.1	.20	142	979	77	531	282
W-2ThO ₂	18.9	.38	173	1193	95	655	356
W-3Re	19.4	.20	214	1475	69	476	249
W-5Re-2ThO ₂	19.1	.20	176	1213	70	483	254
W-24Re-2ThO ₂	19.4	.20	211	1455	50	345	183
W-Hf-C	19.4	.38	207	1427	161	1110	584
W-Re-Hf-C	19.4	.38	314	2165	205	1413	744
Tantalum alloys							
ASTAR 811C	16.9	.51	108	745	84	579	351
Molybdenum alloys							
TZM	10.0	.38	113	779	42	290	295
TZC	10.0	.13	125	862	38	262	267
Niobium alloys							
FS85	10.5	.13	66	455	44	303	295
AS30	9.7	.13	61	421	31	214	224
B88	10.2	.51	77	531	48	331	328

B. 1204° C (2200° F) Data							
Tungsten alloys							
218CS	19.1	0.20	108	745	46	317	170
W-1ThO ₂	19.1	.20	122	841	54	372	198
W-2ThO ₂	18.9	.38	150	1034	70	483	257
W-3Re	19.4	.20	157	1082	46	317	168
W-5Re-2ThO ₂	19.1	.20	148	1020	44	303	160
W-24Re-2ThO ₂	19.4	.20	147	1014	28	193	102
W-Hf-C	19.4	.38	201	1386	111	765	404
W-Re-Hf-C	19.4	.38	281	1937	132	910	480
Tantalum alloys							
ASTAR 811C	16.9	.51	71	490	38	262	157
Molybdenum alloys							
TZM	10.0	.20	77	531	19	131	135
TZC	10.0	.13	79	545	18	124	127
Niobium alloys							
FS85	10.5	.13	40	276	23	159	155
AS30	9.7	.13	33	228	—	—	—
B88	10.2	.51	50	345	28	193	190

TABLE III. - COMPARISON OF FIBER-MATRIX REACTIONS FOR VARIOUS

MATRIX MATERIALS (REF. 15)

Annealing temperature °C	Matrix	No. com- positions investi- gated	Relative no. of cases, percent				
			Recrys- talliza- tion	Inter- metallic compound	Diffu- sion penetra- tion	No. re- crystal- lization	No. re- action
1200	2190	27	93	55	—	7	4
		29	10	83	12	90	10
		30	3	30	—	97	70
1300	2370	27	96	63	—	4	4
		19	21	84	—	79	10
		30	20	80	3	80	13

TABLE IV. - RUPTURE STRENGTHS AND COMPOSITIONS FOR COMPOSITES AND SUPERALLOYS

Ref.	Alloy	Wire	Wire diam		Vol. %	Density		100 hr rupture strength		Stress-density for -100 hr rupture	
			mm	in.		gm/cc	lb/in ³	MN/m ²	ksi	m	in.
30	ZhS6	VRN tungsten	0.3-0.5	0.012-0.020	40	12.5	0.45	138	20	1125	44 300
29	EPD-16	tungsten	0.25	0.010	40	8.3 12.7	0.3 .46	51 131	7.4 19	635 1040	25 000 41 000
31	Nimocast 713C	tungsten	1.27	0.050	20	8.0 10.3	0.29 .37	48 93	7 13.5	613 927	24 000 36 500
19	MARM322E	W-2%ThO ₂	0.08	0.003	40	—	—	48 207	7 30	—	—
9	Ni ₃ Cr ₂ W ₂ Al ₂ Ti ₂	218CS (tungsten)	0.38	0.015	40	9.15 13.3	0.33 .48	23 138	3.3 20	254 1058	10 000 41 700
27		W-2%ThO ₂	0.38	0.015	40	13.0	0.47	193 324	28 47	1513 2491	59 600 98 000
28		W-Hf-C	0.38	0.015	56	12.5	0.45	831 hr rupture strength-242 MN/m ² (35 ksi)		1957	77 000
32	FeCrAlY	W-Hf-C	0.38	0.015	35	11.3	0.41	242	35	2147	84 500
B. Nominal composition of matrix alloys (weight %)											
	ZhS6 EPD-16 Nimocast 713C MARM322E Ni ₃ Cr ₂ W ₂ Al ₂ Ti ₂ FeCrAlY		Ni-12.5Cr-4.8Mo-7W-2.5Ti-5Al Ni-6Al-6Cr-2Mo-11W-1.5Nb Ni-12.5Cr-6Al-1Ti-4Mo-2Nb-2.5Fe Co-21.5Cr-25W-10Ni-0.8Ti-3.5Ta Ni-15Cr-25W-2Ti-2Al Fe-24Cr-5Al-1Y								

TABLE V. - THERMAL CYCLING DATA FOR TUNGSTEN/SUPERALLOY COMPOSITES

Ref.	Composite material	Heat source	Cycle	No. of cycles	Remarks
28	40 v/o W/Nimocast 258	Fluidized bed	RT-1100° C (2010° F)	400	No apparent damage to interface
16	13 v/o W/Nimocast 713C	Fluidized bed	20°-600° C (70°-1110° F) 550°-1050° C (1020°-1920° F) 20°-1050° C (70°-1920° F)	200 2-12 2-25	No cracks Cracks at interface Cracks at interface
42	W/EI435 (14, 24, and 35 v/o)	Electric resistance furnace	RT-1100° C (2010° F) 2.5 min to temp. Water quench	100	No. of cycles for fiber-matrix debonding 14 v/o - 90 to 100 cycles 24 v/o - 60 to 70 cycles 35 v/o - 35 to 50 cycles
43	W/EI435 (15 and 32 v/o)	Self resistance	30 sec to heat and cool 480°-700° C (900°-1290° F) 500°-800° C (930°-1470° F) 530°-900° C (980°-1650° F) 570°-1000° C (1050°-1830° F) 600°-1100° C (1110°-2010° F)	1000	All 15 v/o specimens warped and had a specimen length decrease. Cracks at interface. 35 v/o specimens did not deform externally but matrix cracks between fibers observed.
44	W/NiCrAlY (35 and 50 v/o) W/NiCrAlY (35 and 50 v/o) W/2IDA (35 and 50 v/o) 50 v/o W/NiCrAlY	Self resistance	1 min to heat 4 min to cool RT-1093° C (2000° F)	100	35 v/o warpage and shrinkage 50 v/o no damage
			427°-1093° C (800°-2000° F)	100	35 v/o warpage 50 v/o no damage
				1000	35 v/o warpage and shrinkage 50 v/o no damage
45	30 v/o W-13%ThO ₂ /FeCrAlY	Passage of electric current	1 min to heat 4 min to cool RT-1204° C (2200° F)	1000	No damage Surface roughening but no cracking

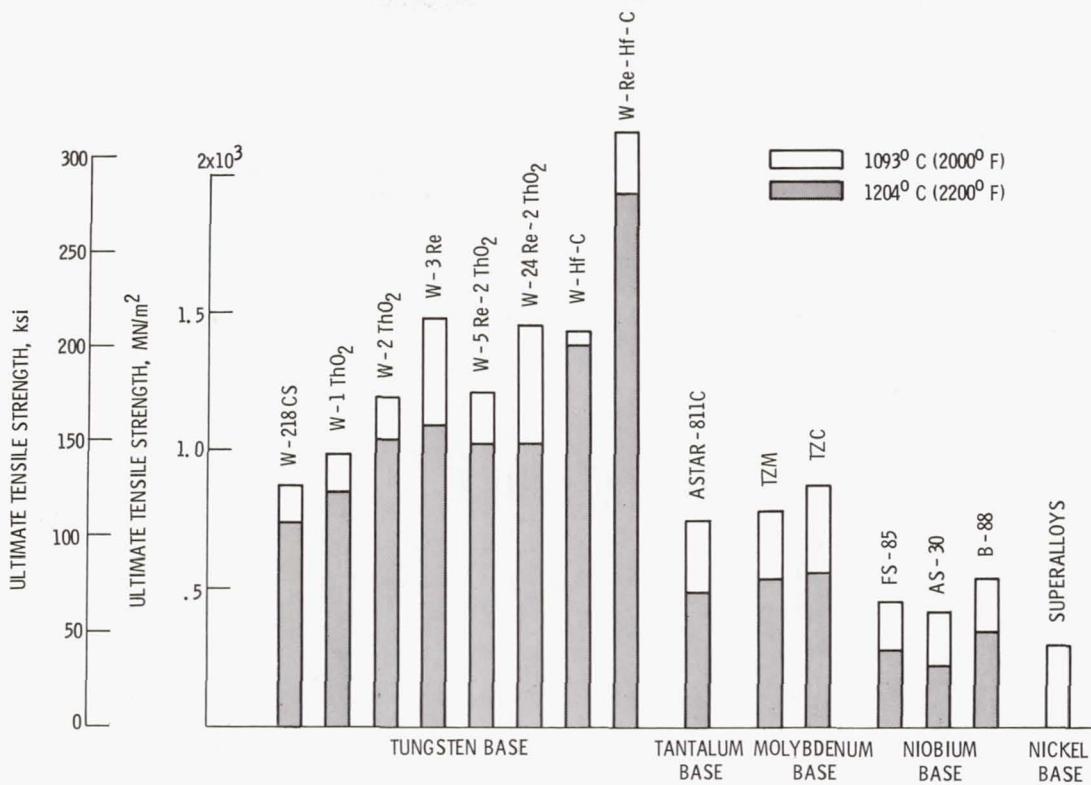


Figure 1. - Ultimate tensile strength for refractory metal wires and superalloys. (Refs. 3 and 4)

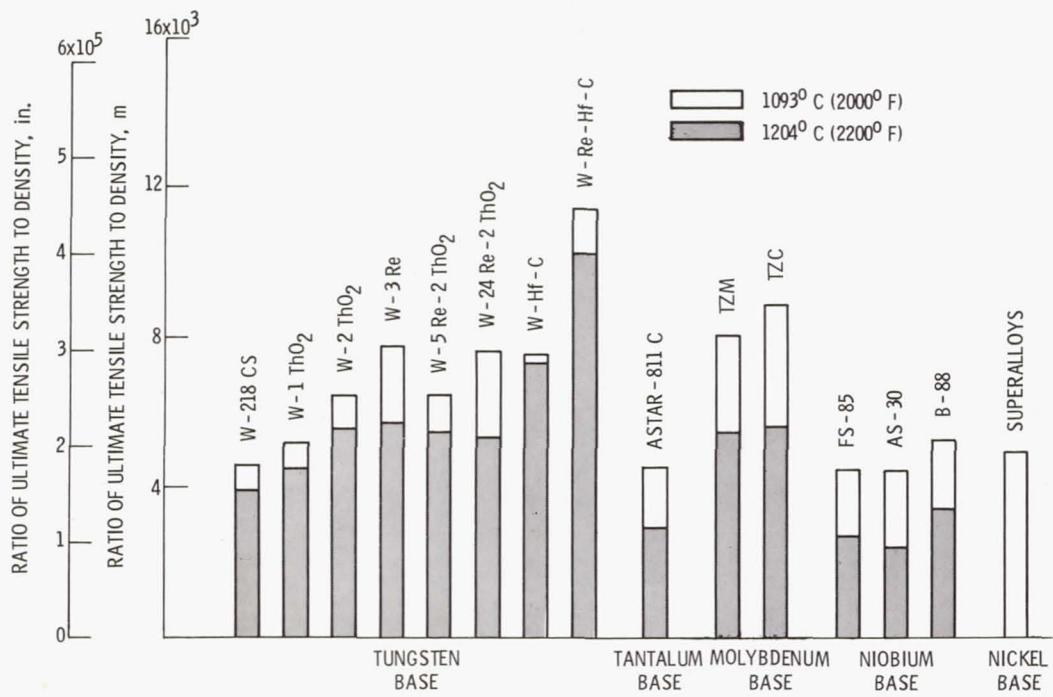


Figure 2. - Ratio of ultimate tensile strength to density for refractory metal wires and superalloys. (Refs. 3 and 4)

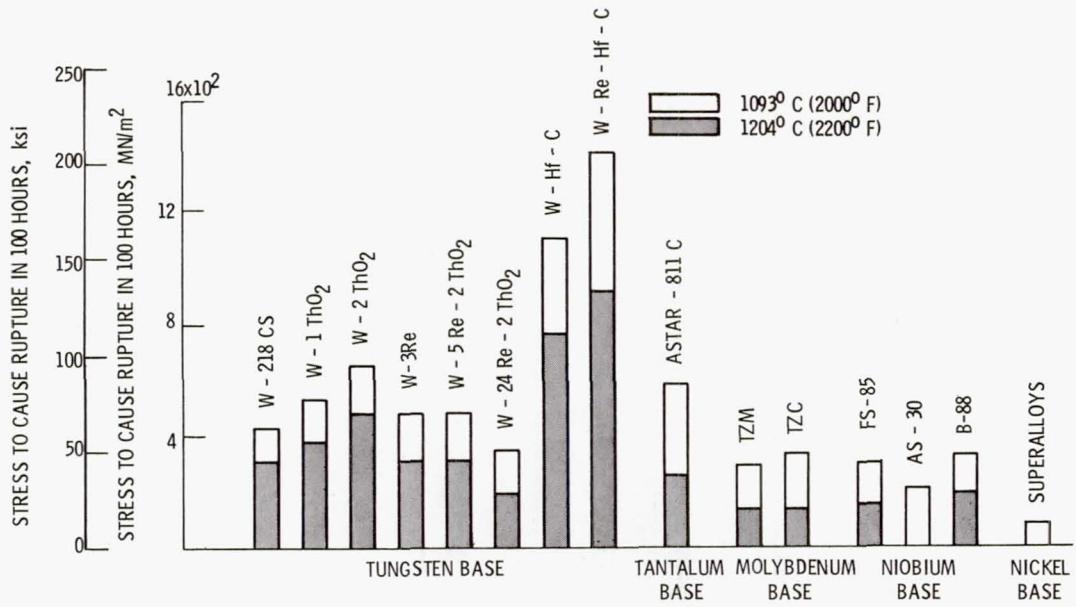


Figure 3. - Stress to cause rupture in 100 hours for refractory metal wires and superalloys. (Refs. 3 and 4)

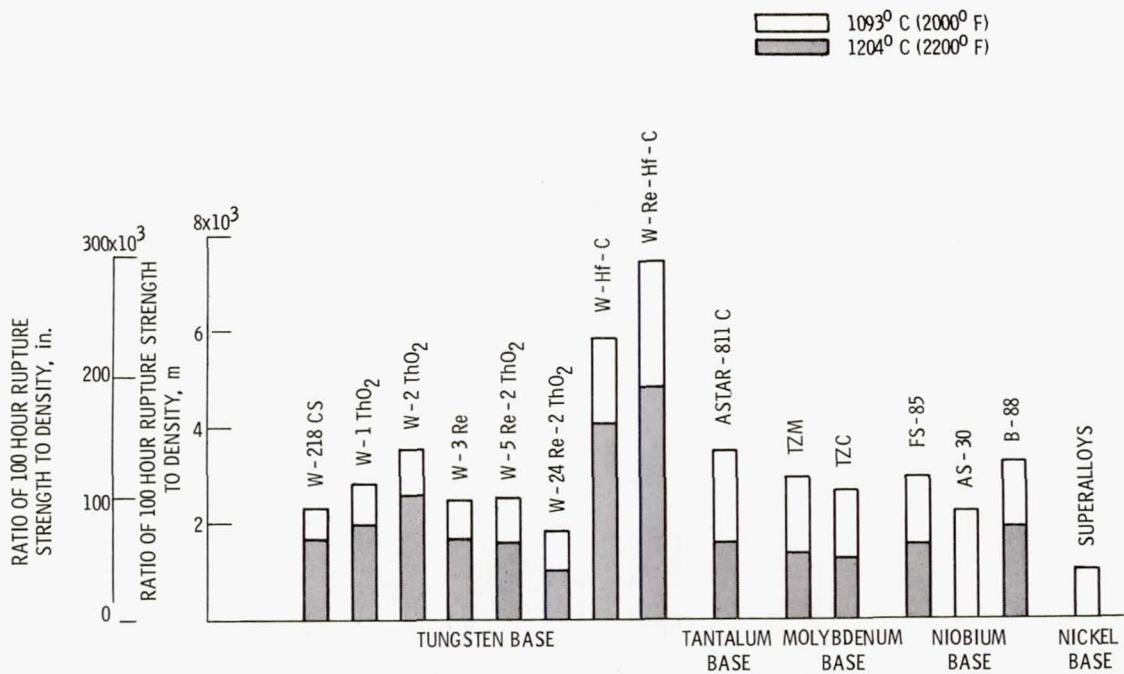


Figure 4. - Ratio of 100 hour rupture strength to density for refractory metal wires and superalloys. (Refs. 3 and 4)

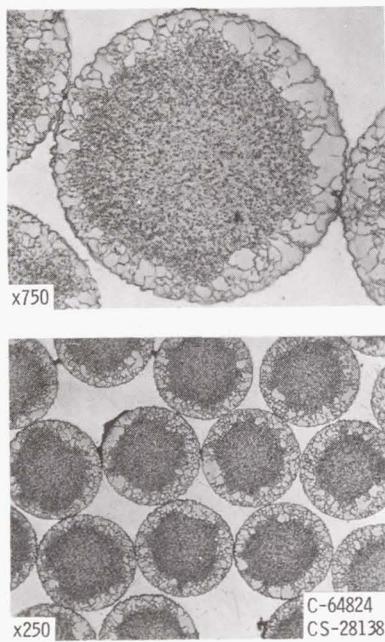


Figure 5. - Recrystallization of tungsten fibers in a copper plus 10 percent nickel matrix. (Ref. 9)

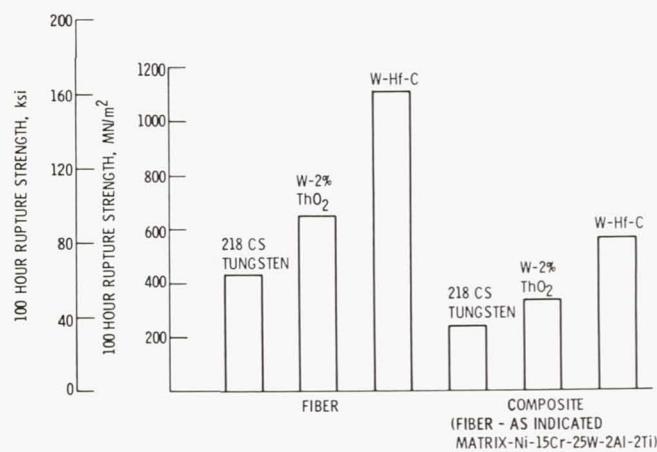


Figure 6. - Comparison of 100 hour rupture strength at 1093° C (2000° F) for fibers and 70 volume percent fiber composites.

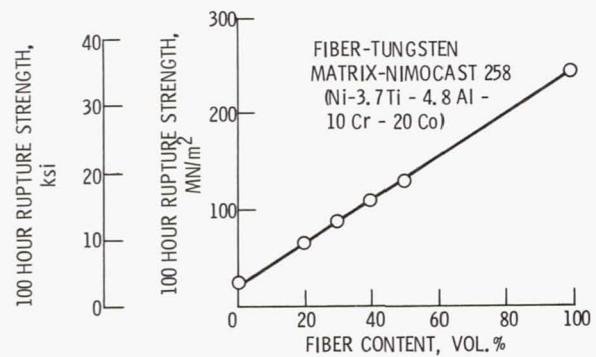


Figure 7. - Effect of fiber content on 100 hour composite rupture strength at 1100° C (2010° F). (Ref. 29)

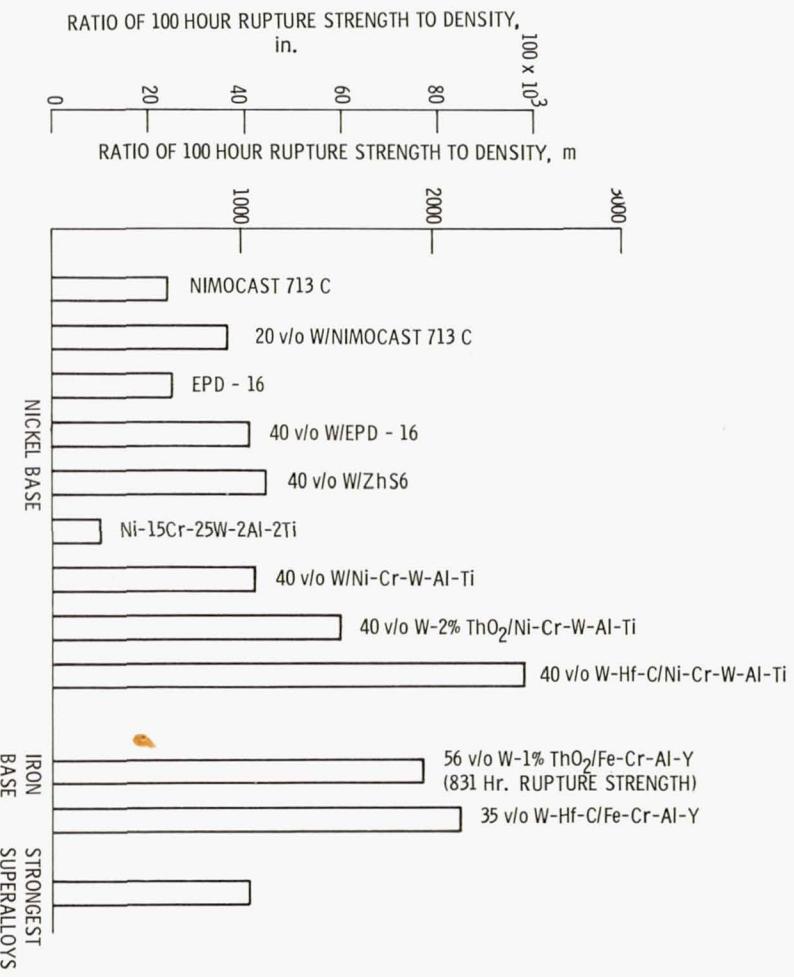


Figure 9. - Comparison of the ratio of 100 hour rupture strength to density for composites and superalloys at 1093° C (2000° F).

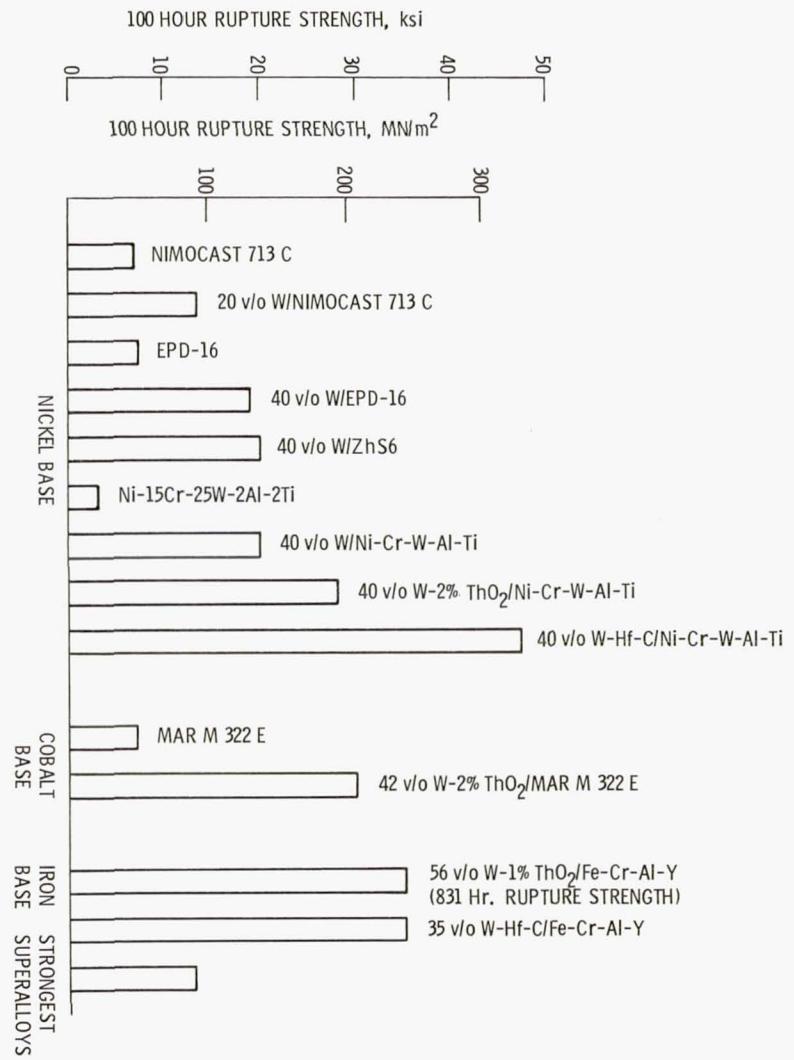


Figure 8. - Comparison of 100 hour rupture strength at 1093° C (2000° F) for composites and superalloys.

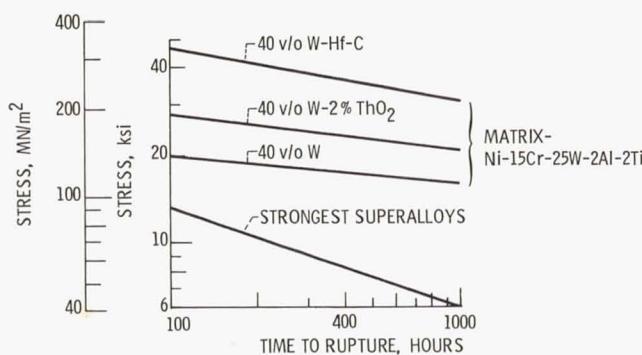


Figure 10. - Stress versus time to rupture comparison for composites and superalloys at 1093° C (2000° F).

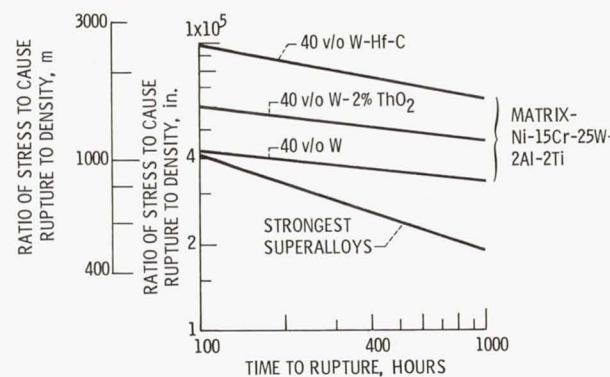


Figure 11. - Stress (to cause rupture) to density ratio for composites and superalloys at 1093° C (2000° F).

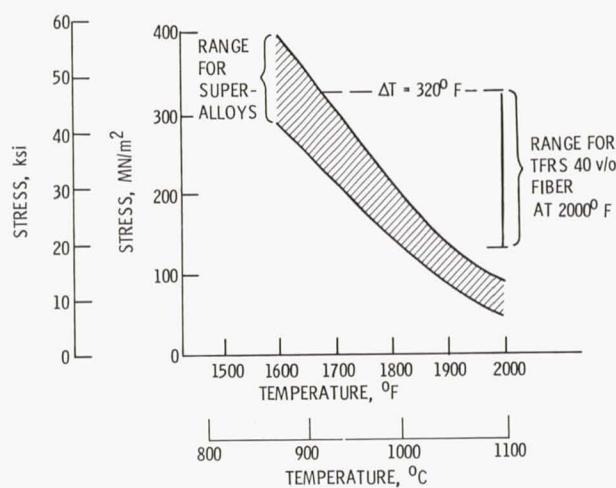


Figure 12. - Comparison of 100 hour rupture strength for TFRS and superalloys.

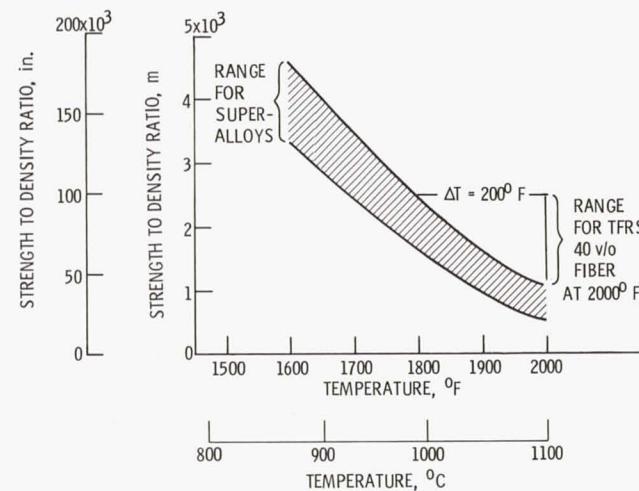


Figure 13. - Comparison of the ratio of 100 hour rupture strength to density for TFRS and superalloys.

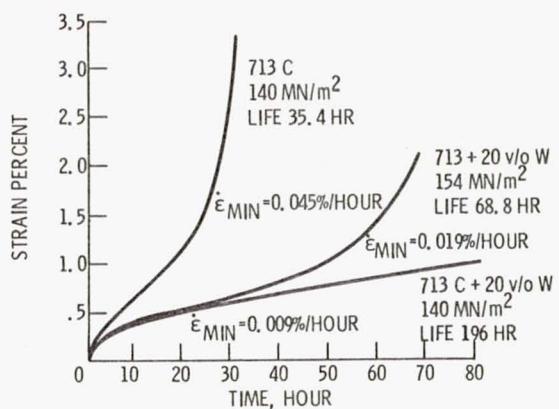


Figure 14. - Comparison of typical creep behaviour of Nimocast 713C with and without tungsten reinforcement at 1100°C (2010°F). (Ref. 31)

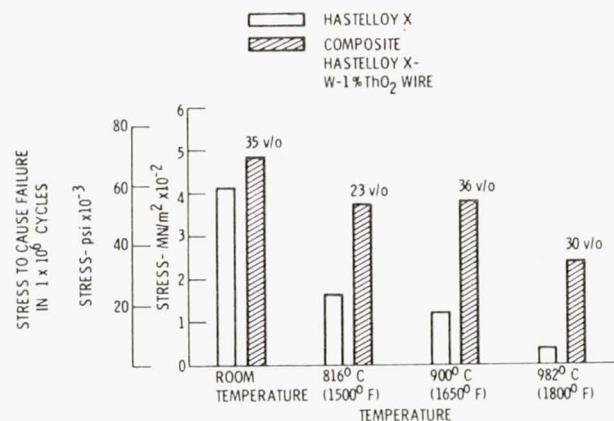


Figure 15. - Stress for failure in 1×10^6 cycles for Hastelloy X and composite. (Ref. 33)

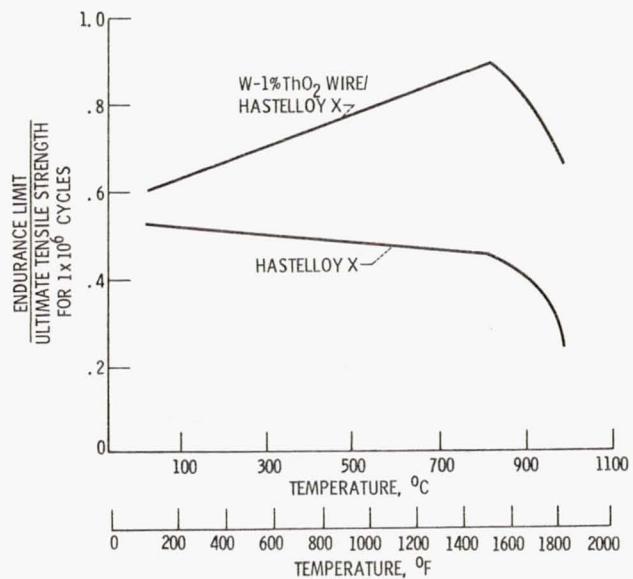


Figure 16. - Ratio of endurance limit to ultimate tensile strength for Hastelloy X and composite tested in axial tension-tension. (Ref. 33)

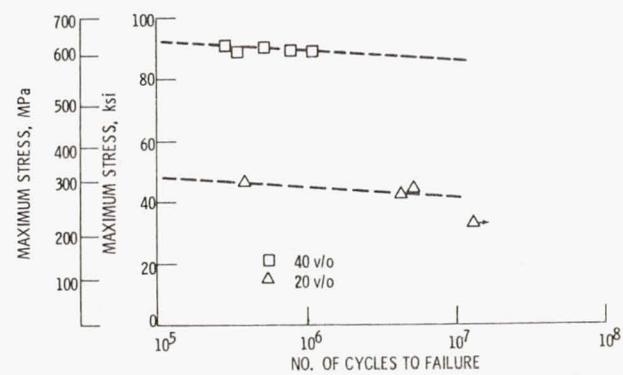


Figure 17. - Stress as a function of number of cycles to failure for W-1%ThO₂/FeCrAlY composites tested at 760°C (1400°F). (Ref. 36)

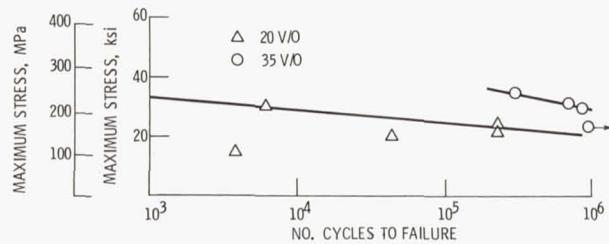


Figure 18. - Stress as a function of number of cycles to failure for W-1%ThO₂/FeCrAlY composites tested at 1038⁰ C (1900⁰ F). (Ref. 36)

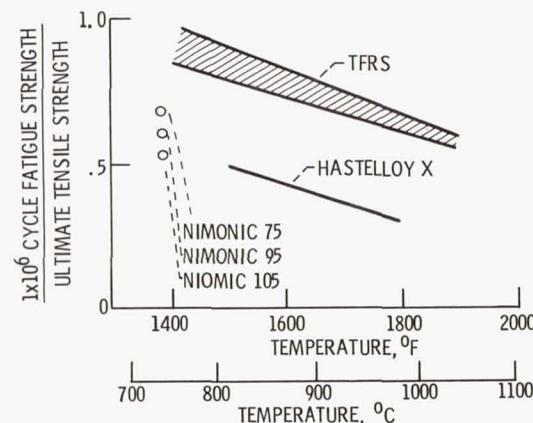


Figure 19. - High-cycle fatigue strength ratio comparison for TFRS and superalloys.

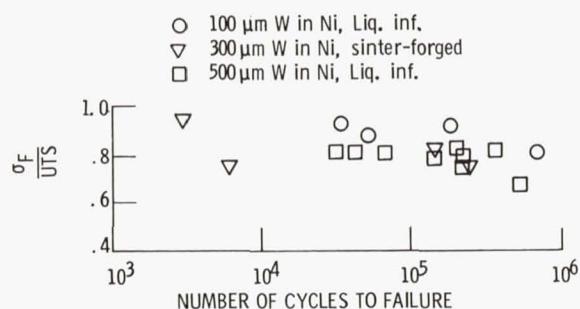


Figure 20. - Ratio of fatigue strength (σ_F) to ultimate tensile strength (UTS) for Ni/W continuously reinforced composites. (Ref. 38)

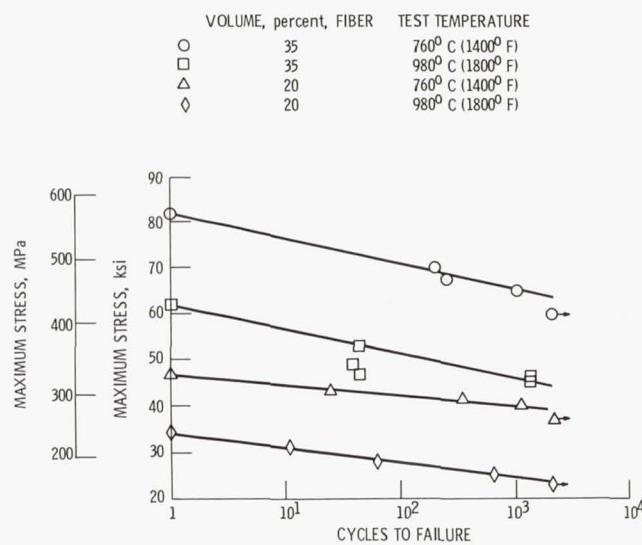


Figure 21. - Maximum stress versus cycles to failure for W-1%ThO₂/FeCrAlY composites. (Ref. 36)

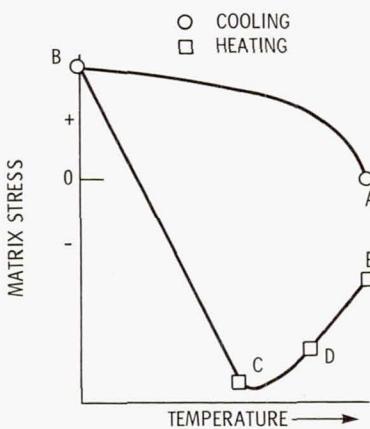
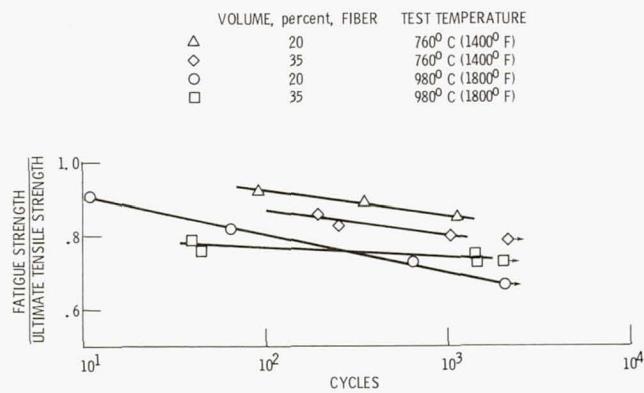


Figure 23. - Matrix stress as a function of heating and cooling.

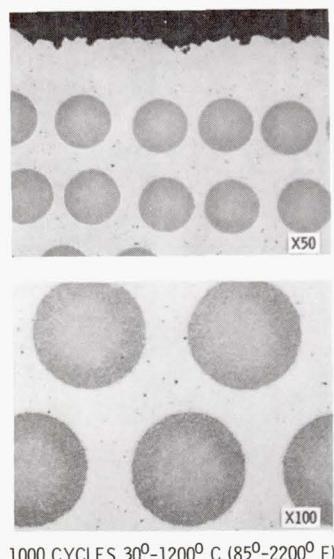


Figure 24. - Photomicrographs of thermally cycled tungsten wire reinforced FeCrAlY composite. (Photos courtesy of Irving Machlin). (Ref. 45)

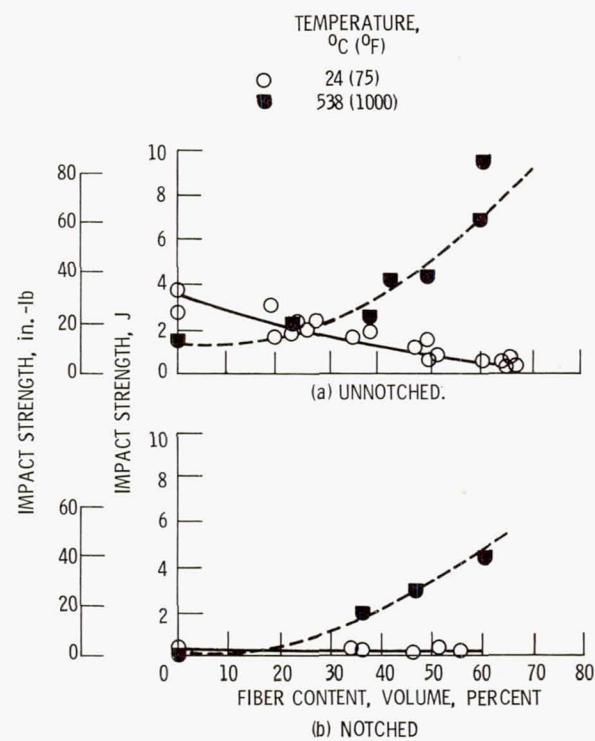


Figure 25. - Impact strength of unnotched and notched as-HIP tungsten/superalloy as a function of fiber content. (Ref. 53)

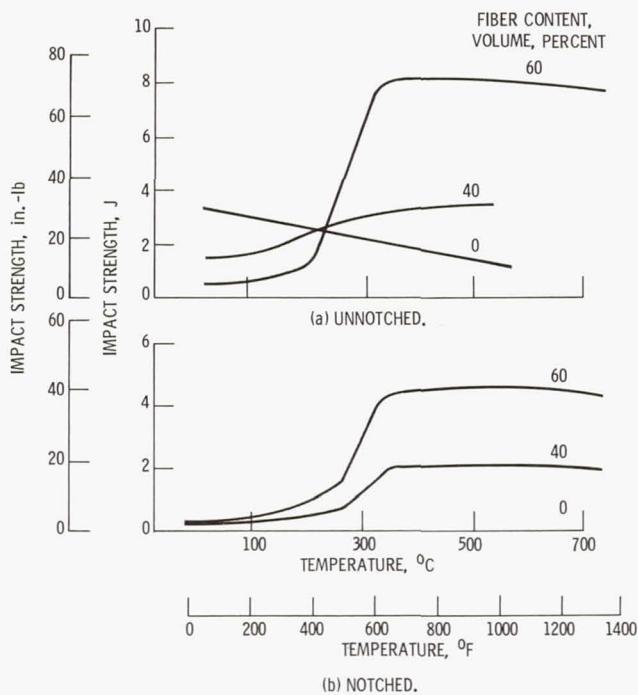


Figure 26. - Impact strength of unnotched and notched tungsten/superalloy as function of temperature and various fiber contents. (Ref. 53)

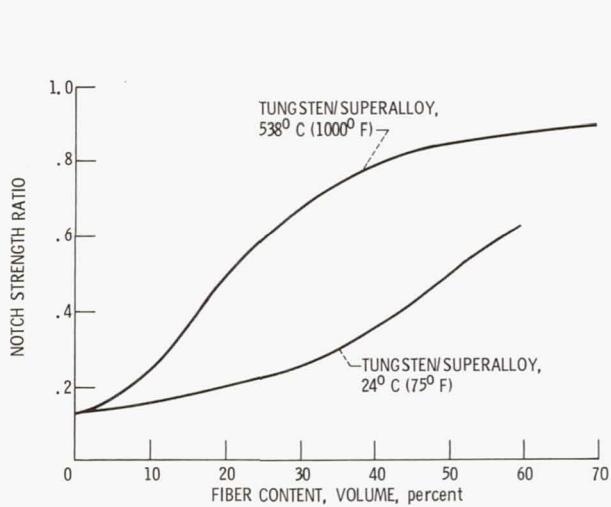


Figure 27. - Notch strength ratio as function of fiber content for tungsten/superalloy composites tested at 24 and 538°C (75°F and 1000°F). (Ref. 53)

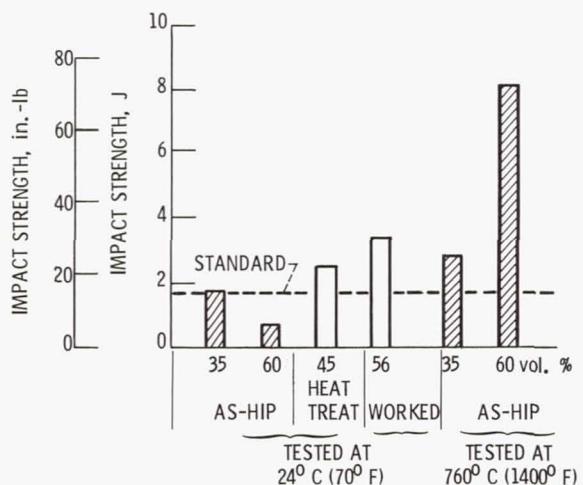


Figure 28. - Miniature Izod impact strengths of unnotched tungsten/nickel-base superalloys compared to minimum impact strength standard used to screen potential turbine blade and vane materials. (Ref. 53)

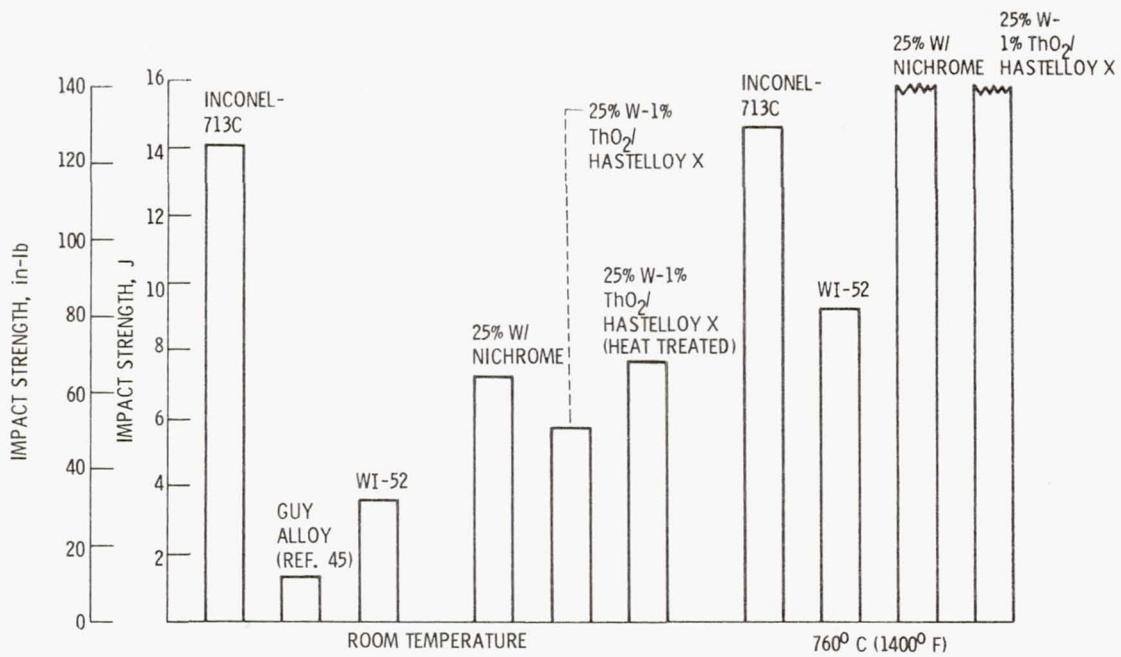
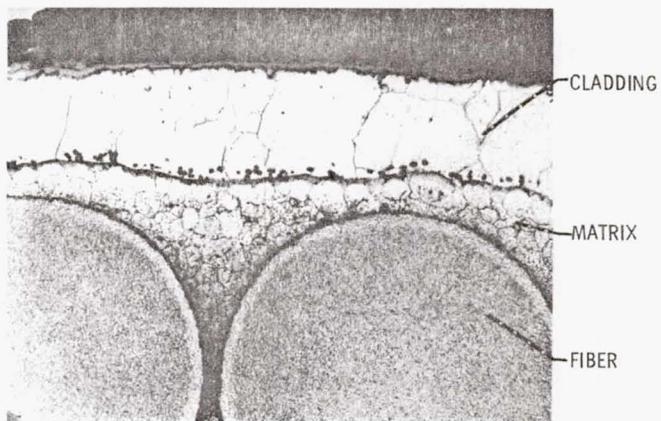


Figure 29. - Miniature Izod impact strength for superalloys and composites.



TEST CONDITION - EXPOSED TO AIR ATMOSPHERE FOR
50 HR AT 2000° F

Figure 30. - Transverse section of oxidized refractory fiber - nickel alloy composite. (Ref. 27)

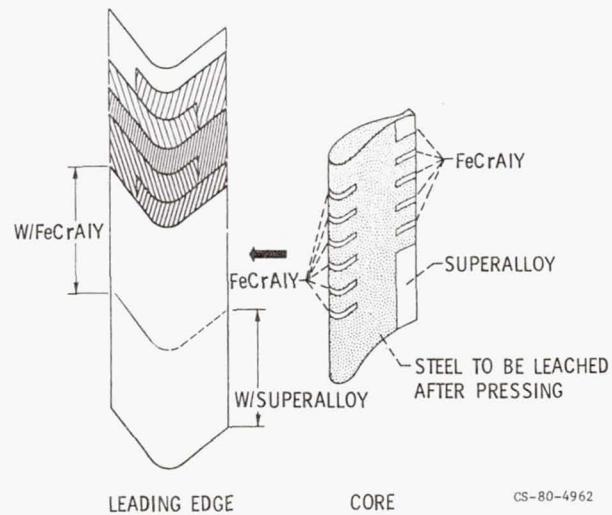


Figure 35. - Schematic of airfoil ply configuration in JT9D blade.

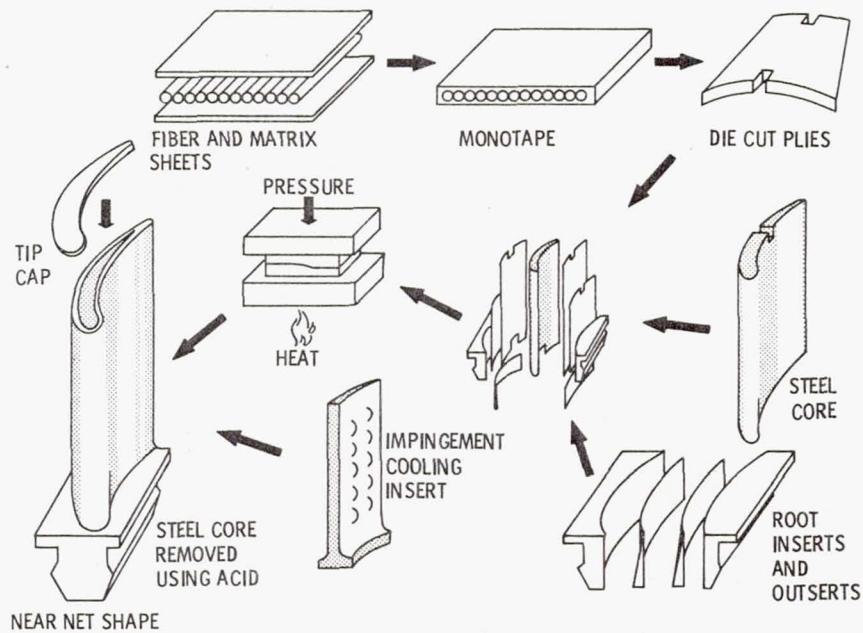


Figure 36. - TFRS blade fabrication process.

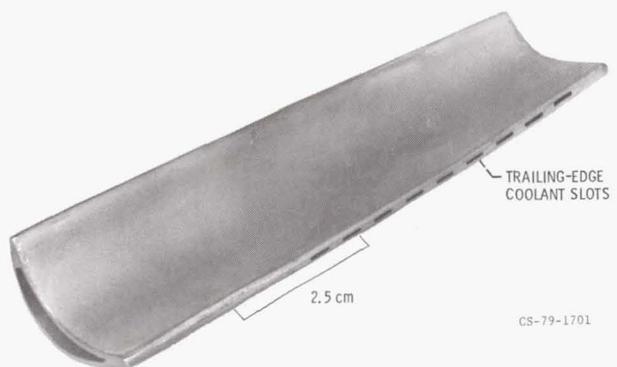


Figure 37. - Composite hollow airfoil.

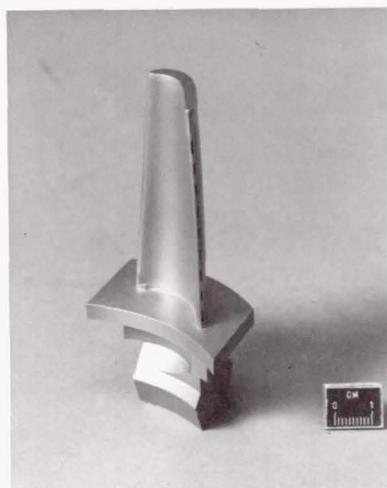


Figure 38. - Hollow composite blade.

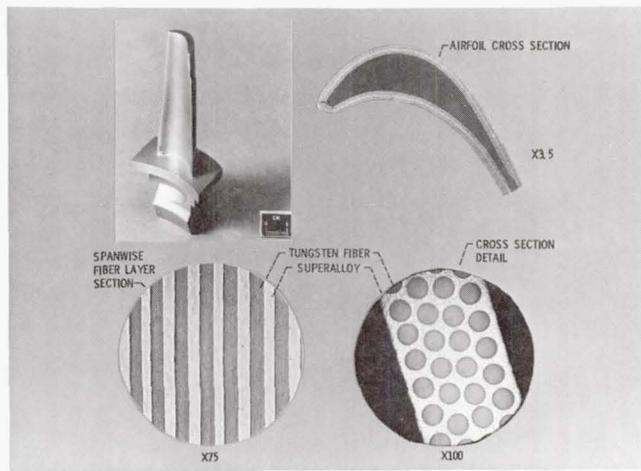


Figure 39. - Tungsten fiber/superalloy composite blade.

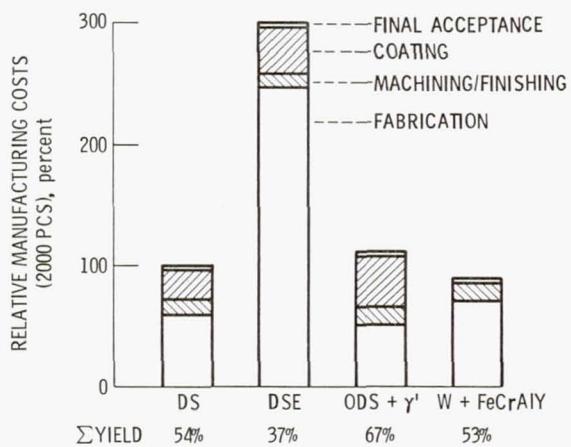


Figure 40. - Cost analysis for JT9D first stage turbine blade. (Ref. 58)

1. Report No. NASA TM-82590	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle TUNGSTEN FIBER REINFORCED SUPERALLOYS - A STATUS REVIEW		5. Report Date	
7. Author(s) Donald W. Petrasek and Robert A. Signorelli		6. Performing Organization Code 505-33-12	
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135		8. Performing Organization Report No. E-837	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546		10. Work Unit No.	
		11. Contract or Grant No.	
		13. Type of Report and Period Covered Technical Memorandum	
		14. Sponsoring Agency Code	
15. Supplementary Notes Prepared for the Fifth Annual Conference on Composites and Advanced Ceramic Materials sponsored by the American Ceramic Society, Merritt Island, Florida, January 18-22, 1981.			
16. Abstract Improved performance of heat engines is largely dependent upon maximum cycle temperatures. Tungsten fiber reinforced superalloys (TFRS) are the first of a family of high temperature composites that offer the potential for significantly raising hot component operating temperatures and thus leading to improved heat engine performance. This status review of TFRS research emphasizes the promising property data developed to date, the status of TFRS composite airfoil fabrication technology, and the areas requiring more attention to assure their applicability to hot section components of aircraft gas-turbine engines.			
17. Key Words (Suggested by Author(s)) Composites; High temperature material; Tungsten alloy fibers; High strength material; Superalloy; Turbine engine material		18. Distribution Statement Unclassified - unlimited STAR Category 24	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages	22. Price*

* For sale by the National Technical Information Service, Springfield, Virginia 22161

National Aeronautics and
Space Administration

Washington, D.C.
20546

Official Business

Penalty for Private Use, \$300

SPECIAL FOURTH CLASS MAIL
BOOK

Postage and Fees Paid
National Aeronautics and
Space Administration
NASA-451



NAS.
Lang
Hampt
Attn

NASA

POSTMASTER: If Undeliverable (Section 158
Postal Manual) Do Not Return
